Quality Assurance/ Sampling and Analysis Project Plan

Sauget Area 1
Dead Creek Sediment Removal Action
Mitigation Plan
Revision 2

Solutia, Inc.
Sauget, Illinois

April 2003

BBL®
BLASLAND, BOUCK & LEE, INC.
engineers & scientists

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QUALITY ASSURANCE/SAMPLING AND ANALYSIS PROJECT PLAN

FOR THE SAUGET AREA DEAD CREEK SEDIMENT REMOVAL ACTION
SOLUTIA INC. – SAUGET, ILLINOIS

Revision Number: 2
Date: April 30, 2003
Prepared By: Blasland, Bouck & Lee, Inc.

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* Conditional Approval

Preface

This SRA Quality Assurance/Sampling and Analysis Project Plan (QA/SAPP) supplements the Sauget Area 1 Dead Creek Sediment Removal Action Mitigation Plan (SRAMP) and presents the sampling and analytical methods and procedures that will be used during implementation of select actions at the site.

This QA/SAPP was prepared in a manner consistent with the following reference and guidance documents:

- United States Environmental Protection Agency's (USEPA's) "Test Methods for Evaluating Solid Waste, SW-846" (USEPA, 1996);
- USEPA's guidance document entitled "EPA Requirements for Quality Assurance Project Plans for Environmental Operations," EPA-QA/R-5 (USEPA, 2001), which replaces QARS-005/80 "Interim Guidance and Specifications for Preparing Quality Assurance Project Plans" (USEPA, 1980);
- USEPA Region 5 guidance document entitled "Region 5 Instructions on the Preparation of a Superfund Division Quality Assurance Project Plan," Revision 0 June 2000; and
- The National Enforcement Investigations Center (NEIC) Policies and Procedures Manual (USEPA, 1991).

Information contained in this QA/SAPP has been organized into the following sections:

Section	Content
Project Management	
1	Project Organization
2	Project Background
3	Project Description
4	Quality Objectives and Criteria for Measurement Data
5	Special Training Requirements/Certification
6	Documentation and Records
Measurement/Data Acquisition	
7	Sampling Process Design
8	Sampling Method Requirements
9	Sample Handling and Custody Requirements
10	Analytical Method Requirements
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12	Instrument/Equipment Testing, Inspection, and Maintenance Requirements
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Details are provided in the subsequent sections. This document also contains pertinent information from the SRAMP related to measuring and evaluating the analytical data.

1. Project Organization

1.1 Project Organization

The Sauget Area 1 Dead Creek Sediment Removal Action (SRA) at the Solutia Inc. facility in Sauget, Illinois will require integration of personnel from the organizations identified below, collectively referred to as the "project team." A detailed description of the responsibilities of each member of the project team is presented below. Figure 1 presents an organization chart for this project outlining lines of authority and lines of communication.

1.1.1 Overall Project Management

On behalf of Solutia Inc. (Solutia), Blasland, Bouck & Lee, Inc. (BBL), has overall responsibility for the SRA activities. BBL personnel will perform related sampling activities. In addition, BBL will evaluate data and prepare the deliverables as specified in the SRA Mitigation Plan (SRAMP). Project direction and oversight will be provided by Solutia. Oversight in the field may also be provided by Solutia. A list of key project management personnel is provided below.

Title	Company/Organization	Name	Phone Number
Technical Manager	Solutia, Inc.	Bruce S. Yare	(314) 674-6370
Remedial Project Manager	Solutia, Inc.	Richard S. Williams	(630) 579-0275
Project Officer	Blasland, Bouck & Lee, Inc.	Alan Fowler	(978) 921-0442
Remedial Project Manager	Blasland, Bouck & Lee, Inc.	David Ludwig	(410) 295-1205
Field Manager	Blasland, Bouck & Lee, Inc.	Todd Merrell	(315) 446-9120
Remedial Project Manager	USEPA	Nabil Fayoumi	(312) 886-6840

1.1.2 Task Manager

The staff performing the investigations and site activities will be directed by representatives of the project team. The personnel responsible for each of the site activities are listed below.

Title	Company/Organization	Name	Phone Number
Sediment Investigation Task Manager	Blasland, Bouck & Lee, Inc.	Steve Truchon	(978) 921-0442
Survey Task Manager	Blasland, Bouck & Lee, Inc.	Tom O'Rourke	(315) 446-9120
Analytical Task Manager	Blasland, Bouck & Lee, Inc.	Laurie A. Indick	(315) 446-9120
Health and Safety Manager	Blasland, Bouck & Lee, Inc.	Jay Keough, C.S.P.	(609) 860-8072

1.1.3 Analytical Laboratory Services

Analytical laboratory services for environmental samples associated with the SRA will be provided by Battelle Marine Sciences Laboratory (methyl mercury) and Brooks Rand Laboratory (total mercury). Laboratory management personnel are listed below.

Title	Company/Organization	Name	Phone Number
Laboratory Director	Battelle Marine Sciences Laboratory	Richard Ecker	(360) 681-3602
Laboratory Remedial Project Manager	Battelle Marine Sciences Laboratory	Brenda LaSorsa	(360) 681-3614
Laboratory Director	Brooks Rand Laboratory	Findlay McKay	(206) 632-6206
Laboratory Remedial Project Manager	Brooks Rand Laboratory	Colin Davies	(206) 632-6206

1.1.4 Quality Assurance/Data Management Staff

The following quality assurance/data management personnel have been assigned to this project:

Title	Company/Organization	Name	Phone Number
Quality Assurance Reviewer	Blasland, Bouck & Lee, Inc.	Laurie A. Indick	(315) 446-9120
Quality Assurance Reviewer	Battelle Marine Sciences Laboratory	Deborah Coffey	(360) 681-3645
Quality Assurance Reviewer	Brooks Rand Laboratory	Frank McFarland	(206) 632-6206
Quality Assurance Reviewer	USEPA	Richard J. Byvik	(312) 886-4071
Database Administrator	Blasland, Bouck & Lee, Inc.	Michael J. Shivell	(315) 446-9120
Data Validator	Blasland, Bouck & Lee, Inc.	Dennis Capria	(315) 446-9120

1.2 Team Member Responsibilities

1.2.1 Solutia, Inc.

Remedial Project Manager

Responsibilities and duties include:

- Provide overall direction of site actions;
- Direct BBL; and
- Review BBL work products, including data, memoranda, letters, reports, and all other documents transmitted to the United States Environmental Protection Agency (USEPA).

1.2.2 Blasland, Bouck & Lee, Inc.

Project Officer
Responsibilities and duties include:

- Oversee BBL work products; and
- Provide BBL approval for major project deliverables.

Remedial Project Manager
Responsibilities and duties include:

- Manage and coordinate the project as defined in the Work Plan, with an emphasis on adhering to the objectives of the site action;
- Review documents prepared by BBL; and
- Assure corrective actions are taken for deficiencies cited during audits of site activities.

Task Managers
The SRA will be managed by Task Managers as set forth in Section 1.1.2. Responsibilities and duties of each Task Manager include:

- Manage relevant day-to-day activities;
- Develop, establish, and maintain files on relevant site activities;
- Review data reductions from the relevant site activities;
- Perform final data review of field data reductions and reports on relevant site activities;
- Assure corrective actions are taken for deficiencies cited during audits of relevant site activities;
- Perform overall Quality Assurance/Quality Control (QA/QC) of the relevant portions of the site activities;
- Review relevant field records and logs;
- Instruct personnel working on relevant site activities;
- Coordinate field and laboratory schedules pertaining to relevant site activities;
- Request sample bottles from laboratory;
- Review the field instrumentation, maintenance, and calibration to meet quality objectives;

- Prepare reports pertaining to relevant site activities; and
- Maintain field and laboratory files of notebooks and logs, data reductions, and calculations, and transmit originals to the Remedial Project Manager.

Field Personnel

Responsibilities and duties include:

- Perform field procedures associated with the investigations as set forth in the Work Plan;
- Perform field analyses and collect QA samples;
- Calibrate, operate, and maintain field equipment;
- Reduce field data;
- Maintain sample custody; and
- Prepare field records and logs.

Quality Assurance Reviewer (QAR)

Responsibilities and duties include:

- Review laboratory data packages;
- Oversee and interface with the analytical laboratory;
- Coordinate field QA/QC activities with Task Managers, including audits of removal action activities, concentrating on field analytical measurements and practices to meet data quality objectives;
- Review field reports;
- Review audit reports;
- Prepare interim QA/QC compliance reports; and
- Prepare a QA/QC report in accordance with United States Environmental Protection Agency (USEPA) guidelines, which includes an evaluation of field and laboratory data and data usability reports.

1.2.3 Battelle Marine Sciences Laboratory and Brooks Rand, LLC

General responsibilities and duties of the analytical laboratories include:

- Perform sample analyses and associated laboratory QA/QC procedures;

- Supply sampling containers and shipping cartons;
- Maintain laboratory custody of sample; and
- Strictly adhere to all protocols in the QA/SAPP.

Remedial Project Manager
Responsibilities and duties include:

- Serve as primary communication link between BBL and laboratory technical staff;
- Monitor workloads and ensure availability of resources;
- Oversee preparation of analytical reports; and
- Supervise in-house chain-of-custody.

Quality Assurance Reviewer
Responsibilities and duties include:

- Supervise the group that reviews and inspects all project-related laboratory activities; and
- Conduct audits of all laboratory activities.

1.2.4 United States Environmental Protection Agency (USEPA)

Remedial Project Manager
Responsibilities and duties include:

- Provide USEPA approval of the Work Plan, supporting documents, and future deliverables; and
- Provide oversight during performance of the site activities.

Quality Assurance Reviewer
Responsibilities and duties include:

- Review and approval of the QA/SAPP;
- Review of the QA/QC portion of any submitted report;

- Monitor progress of the removal actions;
- Ensure that all activities are performed in compliance with applicable federal and regional requirements; and
- Perform field and laboratory audits, if necessary.

2. Project Background

The following summarizes background information for Dead Creek and Borrow Pit Lake. This information was compiled for the *Sauget Area 1 Support Sampling Plan* (O'Brien & Gere, 1999) and the *Sauget Area 1 Dead Creek Sediment Removal Action Mitigation Plan* (Solutia 2002). The applicable Health and Safety Plan for this project was part of the overall project plan entitled "EE/CA and RI/FS Support Sampling Plan, Sauget Area 1" approved by the USEPA on September 9, 1999. BBL's Quality Assurance Manual is provided as Attachment A.

2.1 Site Location and Description

Borrow Pit Lake is located in Sauget and Cahokia, just south of East St. Louis, Illinois (Figure 2). The lake basin is nearly a mile long (5,200 feet) and on average, approximately 500 feet wide. Borrow Pit Lake receives surface waters on its southern end from Dead Creek (Segment F). Lake hydrodynamics are dominated by the Mississippi River flood cycles. Normally, during late summer and winter months of the year, Dead Creek and Borrow Pit Lake are dry with scattered areas of standing water or ice (Woodlot Alternatives, 2001).

2.2 Site History and Summary of Activities

On August 29, 2001, USEPA Region 5 issued an Amended Administrative Order (Docket No. V-W-99-C-554; hereafter, "The Order") for a time-critical sediment removal action in Dead Creek, a 3.5 mile long stream located in Sauget, and Cahokia, St. Clair County, Illinois. As required by the Order, approximately 46,000 cubic yards of impacted sediment were removed from Creek Segments B, C, D, E and F, Site M and the lift station sump where Dead Creek discharges into Old Prairie du Pont Creek. Excavated sediments were transferred to a RCRA and TSCA-compliant containment cell constructed adjacent to the west bank of Dead Creek Segment B just north of Judith Lane. Sediment transfer was completed in January 2002. A temporary plastic cover was installed in the cell to isolate the impacted sediments from storm water. Storm water falling on the cell was contained and treated using granular activated carbon prior to discharge to Creek Segment B.

In addition to installing the cap on the containment cell, Section 3 of the Order, *Work to be Performed*, identified several additional work elements involving characterization of lake sediments, Section 3.C of the Order calls for a Mitigation Plan.

This QA/SAPP specifically addresses the sampling and analysis protocols for a sediment investigation in Borrow Pit Lake to comply with the Order.

3. Project Description

This section describes the investigation activities to be conducted in the Borrow Pit Lake. The two sampling activities that will be performed as part of this investigation are: 1) surface sediment characterization, and 2) sub-surface sediment characterization. No aqueous samples will be collected during this investigation.

Sampling protocols to be followed during the investigation activities are detailed in Section 8 of this QA/SAPP. Samples collected during the investigation will be analyzed in accordance with USEPA Method 1630 (modified for sediment matrix) and USEPA SW-846 Method 7471. Table 1 presents a list of the constituents that will be analyzed for samples collected as part of the Borrow Pit Lake investigation. A description of the sediment sampling effort is presented below.

Sediment sampling in the Borrow Pit Lake is being planned for the winter months of 2003. A timeline for the project is presented in Figure 3. The purpose of implementing the sampling described in this QA/SAPP during these months is to facilitate sampling of the sediment. Sampling during spring or summer would increase the hazards associated with moving around the Borrow Pit Lake. In the presence of surface water, sampling would need to be conducted by boat or wading. In the absence of surface water and during warmer months, sampling would need to be conducted using mud shoes or planking to avoid sinking in the unconsolidated sediment. Sampling during the winter months ensures that the sediments will be sufficiently stabilized as to pose fewer hazards to the field sampling team.

3.1 Sample Location

Coordinates for each sampling location will be established from existing topographic surveys prior to undertaking the sampling effort. This will be accomplished by overlaying a 200 ft by 200 ft grid pattern over the Borrow Pit Lake on the site topographic map. The overlays will result in a series of grid cells that will each have coordinates that will aid in targeting a sample location. A Differential Global Positioning System (DGPS) unit will be used in the field to navigate to each sampling location based on these pre-established coordinates. A field log book will be used to record time, date and other relevant details of the sampling effort (Sections 6 and 16).

Sediment samples will be collected in the Borrow Pit Lake on the 200 ft grid. As directed by the Agency, additional sediment samples may be collected in the Borrow Pit Lake upstream of the confluence of Dead Creek on a 300 ft. grid to evaluate the distribution of mercury in sediments in this backwater area.

Sampling methods, procedures and protocols are the same as those used for the *Sauget Area 1 Support Sampling Plan* (O'Brien & Gere, 1999) and in the *Sauget Area 1 Dead Creek Sediment Removal Action Mitigation Plan* (Solutia 2002). Where appropriate, the procedures and protocols from these plans are summarized in relevant sections of this QA/SAPP.

3.2 Sample Collection

Once located, a sample will be collected from the center of each grid cell at a depth of 0 to 6 inches below ground surface to characterize the biologically active zone, for a total of 60 surface sediment samples (Figure 2). Samples will also be collected at every odd-numbered grid cell (50 percent of the sampling locations) from a depth of 6 inches to the bottom of the sediment profile, which is typically 8 to 15 inches thick, to characterize the sub-surface sediments of the Borrow Pit Lake substrate. Combined, the total number of sediment samples is 90 samples.

Sediment samples will be collected using a manual push-type sediment core sampler. In the event there is ice at the sample location, and ice auger will be used to access sediments with the sampler. The sampler consists of a PVC barrel, polycarbonate (Lexan®) liner, check valve, extension rods, and a "T" handle. A liner will be placed into the bottom of the tube and secured in place. The sampler will then be pushed into the sediment, collecting a sediment sample from 0 to 18 inches below the top of the sediment. Sediment will then be pulled up; creating a slight vacuum that closes the check valve. The tube will be removed from the sampler, and the 0 to 6 inch horizon sectioned accordingly. At odd-numbered grid cells, the remaining sediment (6 to 18 inch) horizon will be collected as a sample. In the event of sampler refusal at less than 18 inches, the total sample depth will be recorded and the sample taken for analysis.

All samples will be prepared and placed into the sample containers in accordance with Standard Operating Procedures (Appendices A and B). Sample containers will be placed on wet ice in coolers. Chain-of-custody procedures will be followed. Copies of chain-of-custody forms, labels, etc. are included for each analytical laboratory in Attachment B. After each sampling location or when all decontaminated sampling equipment has been used, sampling equipment will be decontaminated according to the procedures outlined below.

3.3 Decontamination of Sampling Equipment

The following procedures will be used for sampling equipment requiring decontamination is needed:

- Brush-wash reusable equipment in a bucket or tub using a trisodium phosphate (TSP) or other commercial detergent solution (2 lb of TSP per 10 gal of clean water). Completely brush the entire exterior surface of the article undergoing decontamination. Wash interior wetted surfaces as required.
- Rinse the item with copious quantities of potable water, followed by a distilled water rinse. Rinse reusable sampling equipment used to collect environmental media for metals analysis in a dilute nitric acid solution, followed by a distilled water rinse (per USEPA, 1994; USEPA, 2000; USEPA,2002; ASTM, 1990).
- Air-dry sampling equipment on a clean, non-plastic surface in a well-ventilated, uncontaminated environment. If the sampling device is not to be used immediately, wrap it in aluminum foil and place it in a plastic bag or storage container.

Contain rinse water in a plastic tub with a lid. Empty the contents of this tub daily into a 55 gallon drum located at the IDW storage area.

4. Quality Objectives and Criteria for Measurement Data

The data quality objectives (DQO) process as described in the USEPA Region 5 QAPP Instructions document is intended to provide a "logical framework" for planning field investigations. In particular, the intended uses of the data are specified and appropriate DQOs established. The following sections address in turn each of the seven sequential steps in the Region 5 QAPP DQO process.

Step 1: Problem Statement

Sediments in the Borrow Pit Lake contain mercury. The sampling and analysis program is intended to document the distribution and concentration of total and methyl mercury in the Lake to support risk assessment activities.

Step 2: Decision Identification

The initial use of the data is descriptive (distribution and concentration). The decision has already been made and agreed to by USEPA Region 5 and Solutia that a risk assessment will be undertaken at this site. Thus, no decision process or decision criteria are needed – the threshold decision regarding risk assessment has already been crossed.

Step 3: Identifying Decision Inputs

Decision inputs incorporate both concentration and distribution, and no specific criterion is available for either. However, a fundamental basis for decision making is that a sufficient number of data points be available from the field investigation to support the risk assessment. Thus, the necessary input for the decision is the proportion of non-rejected (usable) data points.

Step 4: Defining Study Boundaries

Study boundaries encompass the BPL sediments and have been developed as a result of intensive discussions between USEPA and Solutia. Figure 2 presents a site location map and shows the locations to be sampled under this QA/SAPP. Sampling will be conducted during a time where Borrow Pit Lake has minimal water overlying the sediments (winter or early spring). Once located, a sample will be collected from the center of each grid cell at a depth of 0 to 6 inches below ground surface to characterize the biologically active zone, for a total of 60 surface sediment samples (Figure 2). Samples will also be collected at every odd-numbered grid cell (50 percent of the sampling locations) from a depth of 6 inches to the bottom of the sediment profile, which is typically 8 to 15 inches thick, to characterize the sub-surface sediments of the Borrow Pit Lake substrate. Combined, the total number of sediment samples is 90 samples.

Step 5: Developing a Decision Rule

As the primary purpose of the data collection is descriptive, and the ultimate decision to undertake a risk assessment has already been made by USEPA Region 5 and Solutia, no decision rule can be specified for the overall study objectives. However, regarding the specific decision input (proportion of non-rejected data, proportion), a decision rule can be devised. Given the large number of samples being collected, some nominal loss of data will not hinder description of the distribution of mercury or decisions regarding the need for risk assessment. Given this, a reasonable decision rule would be that 80% of the data points not be rejected for QA/QC reasons.

The analytical quantitation goals of this QA/SAPP have been designed to ensure that data of adequate quality is collected to support future risk assessment activities. The data collected under this QA/SAPP will be used to estimate exposure to receptor organisms that utilize Barrow Pit Lake (e.g., birds, reptiles, amphibians, and small mammals).

Step 6: Limits on Decision Errors

Specifications for this step call for 1) giving forethought to corrective actions to improve data usability; and 2) understanding the representative nature of the sampling design. This QA/SAPP meets both specifications for this step. Corrective actions are described elsewhere in the document and in appended contractor and laboratory quality management plans. The representative nature of the sampling design has been assured by discussions among Solutia and USEPA. Analytical uncertainty, among other sources of uncertainty, can affect the conclusions of a risk assessment. A description of analytical uncertainty and its effects upon receptor exposure assessment will be included in the Uncertainty Analysis section of the Risk Assessment Report.

Step 7: Design Optimization

In discussions regarding sampling design, USEPA and Solutia considered previous data, data quality objectives, and overall project goals, in keeping with the provisions of this step. In addition, application of the specific decision rules will assure that the findings of the field sampling program are relevant and useful for the specified project purposes. The sampling design was selected to minimize uncertainty in risk assessment conclusions through the use of a representative sampling design with appropriate quality controls.

4.1 Data Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

Screening Data: Screening data affords a quick assessment of site characteristics or conditions. This objective for data quality is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and quality assurance. This objective is generally applied to physical and/or chemical properties of samples, degree of contamination relative to concentration differences, and preliminary health and safety assessment.

Screening Data with Definitive Confirmation: Screening data allows rapid identification and quantitation, although the quantitation can be relatively imprecise. This objective for data quality is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10 percent or more). This objective can also be used to verify less rigorous laboratory-based methods.

Definitive Data: Definitive data are generated using analytical methods, such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both the screening and definitive data categories will be used during the investigation. Field parameters (i.e., turbidity, conductivity, temperature, and pH) that will be obtained during surface water sampling to qualitatively interpret other site data will be determined using screening techniques. All remaining parameters will be determined using definitive techniques.

For this project, three levels of data reporting have been defined. They are as follows:

Level 1 - Minimal Reporting: Minimal or "results only" reporting is used for analyses which, either due to their nature (i.e., field monitoring) or the intended data use (i.e., preliminary screening), do not generate or require extensive supporting documentation.

Level 2 - Modified Reporting: Modified reporting is used for analyses which are performed following standard USEPA-approved methods and QA/QC protocols. Based on the intended data use, modified reporting may require some supporting documentation but not, however, full "CLP-type" reporting.

Level 3 - Full Reporting: Full "CLP-type" reporting is used for those analyses which, based on the intended data use, require full documentation.

The reporting levels for the individual sampling tasks described herein are presented in the following subsections.

4.1.1 Surface Sediment Characterization

Data Use

The sample data will be used for site characterization.

Data Type

Water depth, sediment thickness, and survey data will be collected for all samples. In addition, samples will be collected for laboratory analysis of total and methyl mercury.

Data Quantity

The sample quantities and parametric requirements are summarized in Table 1. Additional information regarding the choice of specific sample collection locations and required analyses can be found in the SRAMP and Section 7 of this document.

Sampling and Analytical Methods

Sampling methods will be as specified in Section 8. The analytical methods are as specified in Section 10. Reporting for total and methyl mercury will be Level 3 (as defined previously).

Measurement Performance Criteria

Precision and accuracy quality control limits for chemical constituents that are used during data review to assess analytical performance are included in Table 2. Quality control limits for field duplicates are also listed in Table 2. Although these quality control limits are only guidelines, frequent failure to meet these limits warrants investigation of the laboratory. Reporting limits are presented in Table 3. The USEPA Region 5 Ecological Data Quality Levels (EDQLs) in sediment and soil for methyl mercury and total mercury (as appropriate) will be analytical quantitation goals. It may not be possible to achieve these goals in many samples, as they approach the technological limits of analytical methods. If necessary for the intended risk assessment application of the data, we will apply surrogate values (proportion of detection limit or statistically derived estimates based on the distribution) in lieu of or in addition to values at the quantitation limit.

Data representativeness is addressed by the sample quantities and locations identified in the Work Plan. Data comparability is intended to be achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of the analytical activities.

4.1.2 Subsurface Sediment Characterization

Data Use

Subsurface sediment data will be used for site characterization.

Data Type

Subsurface sediment from the odd-numbered grid sections will be submitted for laboratory analysis of total and methyl mercury.

Data Quantity

The sample quantities and parametric requirements are summarized in Table 1. Additional information regarding the choice of specific sample collection locations and required analyses can be found in the SRAMP and Section 7 of this document.

Sampling and Analytical Methods

Sampling methods will be as specified in Section 8. The analytical methods are as specified in Section 10. Reporting for total and methyl mercury will be Level 3 (as defined previously).

Measurement Performance Criteria

Precision and accuracy quality control limits for chemical constituents that are used during data review to assess analytical performance are included in Table 2. Quality control limits for field duplicates are also listed in Table 2. Although these quality control limits are only guidelines, frequent failure to meet these limits warrants investigation of the laboratory. Reporting limits are presented in Table 3. The USEPA Region 5 EDQLs in sediment for methyl mercury and total mercury will be analytical quantitation goals.

Data representativeness is addressed by the sample quantities and locations identified in the Work Plan. Data comparability is achieved through the use of standard USEPA-approved methods. Data completeness will be assessed at the conclusion of analytical activities.

5. Special Training Requirements/Certification

In compliance with the Occupational Safety and Health Administration's (OSHA) final rule, "Hazardous Waste Operations and Emergency Response," 29CFR§1910.120(e), all personnel performing SRA activities at the site will have completed the requirements for OSHA 40-hour Hazardous Waste Operations and Emergency Response training. Persons in field supervisory positions will have also completed the additional OSHA 8-hour Supervisory Training.

6. Documentation and Records

6.1 General

Sediment samples will be collected as described in the SRAMP and Section 8 of this document. Detailed descriptions of the sample designation system, documentation and reporting requirements are presented below.

6.1.1 Sample Designation System

6.1.2 Sample Codes

The sample designation code will provide each sample with a unique "name." This alphanumeric system will apply to all samples that are to be transmitted to Battelle and Brooks Rand for analysis. The format was chosen to be consistent with existing sample designation codes in the project database. The designation code system includes a three-letter prefix indicating the sample location (BPL – Borrow Pit Lake) and a five-digit code indicating the chemical analyte and sample matrix (HGSED – Mercury Sediment Sample), followed by a two-digit sample grid number, and an indicator of sample depth (0-0.5 FT or 0.5 FT to bottom depth of sample FT). The two-digit code, which designates the sample grid number, is divided as follows:

- 01 through 60.
- 61 through 70 reserved for field duplicates.
- 71 through 80 reserved for rinse blanks.

Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis for both total and methyl mercury analysis will be noted on the chain-of-custody forms, and the associated additional sample containers will be labeled with the appropriate suffix (MS or MSD). Field duplicate will be designated, sequentially, with grid numbers 61 through 70 and shall otherwise be in no way distinguishable by the laboratory as duplicate samples. Rinse blanks will be numbered, sequentially, with grid numbers 71 through 80.

6.2 Field Documentation

Field personnel will provide comprehensive documentation covering various aspects of field sampling, field analysis, and sample chain-of-custody. This documentation constitutes of a record that allows reconstruction of field events to aid in the data review and interpretation process. Documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the action include:

- Daily Production Documentation - A field notebook consisting of a waterproof, bound notebook that will contain a record of all activities performed at the site.

- Sampling Information - Detailed notes will be made as to the exact sampling location, physical observations, and weather conditions (as appropriate).

- Sample Chain-of-Custody - Chain-of-custody (COC) forms will provide the record of responsibility for sample collection, transport, and submittal to the laboratory. COC forms will be filled out at each sampling site, at a group of sampling sites, or at the end of each day of sampling by BBL's field personnel responsible for sample custody. In the event that the samples are relinquished by the designated sampling person to other sampling or field personnel, the COC form will be signed and dated by the appropriate personnel to document the sample transfer. The original COC form will accompany the samples to the laboratory, and copies will be forwarded to the project files. Copies of COC forms are included in Attachment A.

  Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

6.3 Laboratory Documentation Files

6.3.1 Laboratory Project Files

Battelle Marine Sciences Laboratory and Brooks Rand, LLC will establish files for pertinent data. The files will include correspondence, faxed information, phone logs, and COC forms. The laboratories will retain project files and data packages for not less than a period of 5 years.

6.3.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of each laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded on laboratory forms or personal notebook pages by the analyst. These notes will be dated and will also identify the analyst, the instrument used, and the instrument conditions.

Laboratory notebooks will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance with this QA/SAPP. All entries and calculations will be verified by the laboratory group leader. If all entries on the pages are correct, then the laboratory group leader will initial and date the pages. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

6.3.3 Computer Tape and Hard Copy Storage

All electronic files and deliverables will be maintained on magnetic tape or disk for not less than five years; hard copy data packages (or electronic copies) will be maintained in the files for five years.

6.4 Data Reporting Requirements

6.4.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the SRAMP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

Where appropriate, field data forms and calculations will be processed and included in appendices to a Site Action Report (when generated). The original field logs, documents, and data reductions will be kept in the project file at the BBL office in Syracuse, New York.

6.4.2 Laboratory Data Reporting

The laboratories are responsible for preparing full CLP-equivalent data packages for all total and methyl mercury data, reduced data packages, and case narratives for all other analyses.

Data reports for all parameters will include, at a minimum, the following items:

Narrative: Summary of activities that took place during the course of sample analysis, including the following information:

- Laboratory name and address;
- Date of sample receipt;
- Cross reference of laboratory identification number to contractor sample identification;
- Analytical methods used;
- Deviations from specified protocol; and
- Corrective actions taken.

Included with the narrative will be any sample handling documents, including field and internal COC forms, air bills, and shipping tags.

Analytical Results: Reported according to analysis type and including the following information, as acceptable:

- Sample ID;
- Laboratory ID;
- Date of collection;
- Date of receipt;
- Date of extraction;
- Date of analysis; and

- Detection limits.

Sample results on the report forms will be corrected for dilutions. Sediment samples will be reported on a dry weight basis. Unless otherwise specified, results will be reported uncorrected for blank contamination.

The data for total and methyl mercury analyses will be expanded to include supporting documentation necessary to provide a CLP-equivalent package. This additional documentation will include, but is not limited to, raw data required to recalculate any result, including instrument printouts and quantitation reports. The report also will include standards used in calibration and calculation of analytical results; sample extraction, digestion, and other preparation logs; standard preparation logs; instrument run logs; and moisture content calculations. In addition to those items mentioned previously, the laboratories are required to report the following:

- Sample analysis summary data sheets;
- Initial and continuing calibration summary sheets;
- Detection limit standard recovery summary;
- Preparation and calibration blank summary;
- Matrix spike and matrix spike duplicate summary;
- Laboratory control sample recovery summary;
- Instrument detection limits;
- Preparation logs;
- Analysis logs; and
- All raw data associated with the analyses.

6.5 Project File

Project documentation will be placed in a single project file at the BBL office in Syracuse, New York. This file will consist of the following components:

1. Agreements (filed chronologically);
2. Correspondence (filed chronologically);
3. Memos (filed chronologically); and
4. Notes and data (filed by topic).

Reports (including QA reports) will be filed with correspondence. Analytical laboratory documentation (when received) and field data will be filed with notes and data. Filed materials may be removed and signed out by authorized personnel on a temporary basis only.

7. Sampling Process Design

The sampling process for the work described in the SRAMP is based on a grid sampling design. The number of samples was determined through selecting a grid size that would spatially represent an adequate proportion of the Borrow Pit Lake substrate. The size of each grid for the Borrow Pit Lake sediment characterization is 200 feet by 200 feet. Additional sampling in segments of Dead Creek will be conducted every 300 feet as needed. This design results in 60 surface samples and 30 subsurface samples (plus field duplicates) that cover an area of 5,200 feet by 500 feet.

Prior to sampling a basemap of the grid overlay on to Borrow Pit Lake will be used by a BBL survey team to place grade stakes at locations where grids bisect. This survey will allow the sediment sampling team to identify approximate locations within each grid where sediment samples will be collected. As mentioned in Section 3, the actual location of samples will be recorded with DGPS.

8. Sampling Method Requirements

As part of the field investigations, several standard field procedures will be performed. They include:

- Sediment core collection and characterization;
- Surface sediment sampling; and
- Subsurface sediment sampling.

This section of the QA/SAPP introduces and references the appropriate detailed procedure in the Appendices to this QA/SAPP. Included are the ancillary procedures for equipment cleaning. Sample handling, packing, and shipping are discussed in Section 9. Sample quantities and analytical constituents and parameters for the following sections are summarized in Table 1. The required sample containers, volumes, preservation, and holding times are summarized in Table 4.

8.1 Sampling Equipment and Procedures

8.1.1 Sediment Sampling

Sediment samples will be collected utilizing clear Lexan® tubing. Probing and sediment sampling activities will be conducted utilizing the materials and procedures provided in Appendix A. Sediment samples will be collected at discrete intervals and analyzed, as discussed in Section 8.1.3. The sediment samples will be shipped to the laboratory following the protocols set forth in Appendix B. Equipment will be thoroughly cleaned between sample locations as described in Appendix C. Materials and wastes (i.e., sediment, water, disposable equipment, etc.) generated during implementation of the SRA will be collected and disposed of appropriately, as discussed in Section 9.3.

8.1.2 Survey of Sample Locations

Sample locations and transect end points will be surveyed and documented so that they can be located at a later date, if necessary. Conventional surveying equipment and techniques combined with Global Positioning System (GPS) technology will be used to tie the locations to permanent reference points. The sample locations and cross-section delineations will be incorporated into a base map that will be used for future sampling and presentations.

8.1.3 Sediment Characterization

At each location, sediment probing will be conducted with metal rods and hand-coring equipment. Soft sediment areas penetrable by a metal rod will be considered sediment deposits and will be sampled with a clear Lexan® tube for visual inspection. The Lexan® tubing will be hand-driven until refusal. Each core will be described and distinct strata within the cores will be classified according to the Unified Soil Classification System (USCS). In addition to visual inspection and description, cores will be photographed along with an identification sheet. All locations will be surveyed using conventional ground survey techniques or GPS technology.

For each sample collection point, the following information will be obtained and recorded:

- Surveyed location;
- Depth of sediment (depth of refusal);
- Depth of water;
- Description of the bank slope and condition (with respect to erosion);
- USCS description(s) of sediment layer(s);
- Secondary sediment descriptions (i.e., color, odor, presence of debris, types of materials);
- Physical features of the river, including a description of the river bottom; and
- Other appropriate field conditions and observations.

9. Sample Handling and Custody Requirements

9.1 Sample Containers and Preservation

Appropriate sample containers, preservation methods, and laboratory holding times for SRA samples are shown in Table 4.

The analytical laboratories will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased pre-cleaned according to USEPA Office of Solid Waste and Emergency Response (OSWER) Directive 9240.05A requirements. The field personnel will be responsible for properly labeling containers and preserving samples (as appropriate). Sample labeling procedures are described in Appendix B.

9.2 Field Custody Procedures

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through time of transport to the analytical laboratory. Persons will have "custody of samples" when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field COC forms.

9.2.1 Field Logbooks

Field logbooks will provide the means of recording data collecting activities performed. As such, entries will be described in as much detail as possible so that persons going to the site could re-construct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure location when not in use. Each logbook will be identified by the project-specific document number. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned;
- Logbook number;

- Project name;
- Project start date; and
- End date.

Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station shall be recorded. The number of the photographs taken of the station, if any, will also be noted.

Samples will be collected following the sampling procedures documented in Section 8. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

9.2.2 Sample Labeling

Preprinted sample labels will be affixed to sample bottles prior to delivery at the sampling site. The following information is required on each sample label:

- Project;
- Date collected;
- Time collected;
- Location;
- Sampler;
- Analysis to be performed;
- Preservative; and
- Sample number.

9.2.3 Field Chain of Custody Forms

Completed COC forms will be required for all samples to be analyzed. Sample COC forms are provided in Attachment B. COC forms will be initiated by the sampling crew in the field. The COC forms will contain the unique sample identification number, sample date and time, sample description, sample type, preservation (if any), and analyses required. The original COC form will accompany the samples to the laboratory. Copies of the COC will be made prior to shipment (or multiple copy forms used) for field documentation. The COC forms will remain with the samples at all times. The samples and signed COC forms will remain in the possession of the sampling crew until the samples are delivered to the express carrier (e.g., Federal Express) or hand delivered to a mobile or permanent laboratory, or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink unless prohibited by weather conditions. The labels will include sample information such as: sample number and location, type of sample, date and time of sampling, sampler's name or initials, preservation, and analyses to be performed. The completed sample labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the facility or agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

9.3 Management of Investigation Derived Materials and Wastes

Disposable equipment, debris and decontamination rinsate (e.g., distilled water containing small amounts of solvent) will be containerized during the sampling events and labeled for appropriate disposal.

9.4 Packing, Handling and Shipping Requirements

Sample packaging and shipment procedures are designed to insure that the samples will arrive at the laboratory, with the COC, intact.

Samples will be packaged for shipment as outlined below:

- Ensure that sample containers have the sample labels securely affixed to the container with clear packing tape;
- Check the caps on the sample containers to ensure that they are properly sealed;
- Wrap the sample container cap with clear packing tape to prevent it from becoming loose;
- Complete the COC form with the required sampling information and ensure that the recorded information matches the sample labels. NOTE: If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the COC prior to this transfer. The appropriate personnel will sign and date the COC form to document the sample custody transfer;
- Using duct tape, secure the outside drain plug at the bottom of the cooler;
- Wrap sample containers in bubble wrap or other cushioning material;
- Place 1 to 2 inches of cushioning material at the bottom of the cooler;
- Place the sealed sample containers into the cooler;
- Place ice in plastic bags and seal. Place loosely in the cooler;
- Fill the remaining space in the cooler with cushioning material;
- Place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid;
- Close the lid of the cooler, lock, and secure with duct tape;
- Wrap strapping tape around both ends of the cooler at least twice; and
- Mark the cooler on the outside with the following information: shipping address, return address, "Fragile" labels, and arrows indicating "this side up." Cover the labels with clear plastic tape. Place a signed custody seal over the cooler lid.

Samples will be packaged by the field personnel and transported as low-concentration environmental samples. The samples will be hand-delivered or delivered by an express carrier within 48 hours of the time of collection. Shipments will be accompanied by the COC form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the COC form as long as the forms are sealed inside the sample cooler and the custody seals remain intact.

Sample custody seals and packing materials for filled sample containers will be provided by the analytical laboratories. Example seals are provided in Attachment B. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to eliminate the possibility of container breakage.

Additional procedures for packing, handling, and shipping environmental samples are included in Appendix B.

9.5 Laboratory Custody Procedures

9.5.1 General

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field COC form will accompany all samples requiring laboratory analysis. The laboratory will use COC guidelines described in the USEPA guidance documents. Samples will be kept secured in the laboratory until all stages of analysis are complete. All laboratory personnel having samples in their custody will be responsible for documenting and maintaining sample integrity.

9.5.2 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will verify the package seal, open the package, and compare the contents against the field COC. If a sample container is received broken, the sample is in an inappropriate container, or has not been preserved by appropriate means, BBL will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be recorded in the laboratory tracking system. Relevant custody documentation will be placed in the project file.

9.5.3 Sample Analysis

Analysis of an acceptable sample will be initiated by worksheets that contain all pertinent information for analysis. The analyst will sign and date the laboratory COC form when removing the samples from storage.

Samples will be organized into sample delivery groups (SDGs) by the laboratory. A SDG may contain up to 20 field samples (field duplicates, trip blanks, and rinse blanks are considered field samples for the purposes of SDG assignment). All field samples assigned to a single SDG shall be received by the laboratory over a maximum of 7 calendar days, and must be processed through the laboratory (preparation, analysis, and reporting) as a group. Every SDG must include a minimum of two site-specific matrix spike/matrix spike duplicate (MS/MSD) pair, which shall be received by the laboratory at the start of the SDG assignment.

Each SDG will be self-contained for all of the required quality control samples. All parameters within an SDG will be extracted and analyzed together in the laboratory. These rules for analysis will ensure that the QC samples for an SDG are applicable to the field samples of the same SDG and that the best possible comparisons may be made.

9.5.4 Sample Storage Following Analysis

Samples will be maintained by the laboratory for one month after the final report is delivered to BBL. After this period, the laboratory is responsible for the disposal of the samples. Unused portions of the samples, samples extracts and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations as specified in their Standard Operating Procedure for waste disposal (See Attachment B).

10. Analytical Method Requirements

10.1 Laboratory Parameters and Methods

The methods listed below include the range of analyses expected to be performed. Standard operating procedures (SOPs) for these analyses are provided in Attachment B.

10.1.1 Sediment Samples

Surface sediment samples from all grids and subsurface sediment samples from the odd-numbered grids will be analyzed for:

Total Mercury	USEPA SW-846 Method 7471
Methyl Mercury	USEPA Method 1630 (modified for sediment matrix)

11. Quality Control Requirements

11.1 Quality Assurance Indicators

The overall quality assurance objective for this QA/SAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal quality control, audits, preventive maintenance, and corrective action, such that valid data will be generated. These procedures are presented or referenced in the following sections of the QA/SAPP. Specific QC checks are discussed in Section 11.2.

Quality assurance indicators are generally defined in terms of five parameters:

1. Representativeness;
2. Comparability;
3. Completeness;
4. Precision; and
5. Accuracy.

Each parameter is defined below. Specific objectives for the site actions are set forth in other sections of this QAPP as referenced below.

11.1.1 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the site. The actions have been designed to assess the presence of the chemical constituents at the time of sampling. The Site SRAMP presents the rationale for sample quantities and location. This QA/SAPP presents field sampling methodologies and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

11.1.2 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between phases of the actions (if additional phases are required) will be maintained through consistent use of the sampling and analytical methodologies set forth in this QA/SAPP and through the use of established QA/QC procedures, and the utilization of appropriately trained personnel.

11.1.3 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 11.6.

11.1.4 Precision

Precision is a measure of the reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the objectives of the action. To maximize precision, sampling and analytical procedures will be followed. All work for the site actions will adhere to established protocols presented in the QA/SAPP. Checks for analytical precision will include the analysis of matrix spike, matrix spike duplicates, laboratory duplicates and field duplicates. Checks for field measurement precision will include obtaining duplicate field measurements. Further discussion of precision QC checks is provided in Section 11.4.

11.1.5 Accuracy

Accuracy is a measure of how close a measured result is to the true value. Both field and analytical accuracy will be monitored through initial and continuing calibration of instruments. In addition, reference standards, matrix spikes, blank spikes, and surrogate standards will be used to assess the accuracy of the analytical data. Further discussion of accuracy checks is provided in Section 11.5.

11.2 Field Quality Control Checks

11.2.1 Sample Containers

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary. The bottles will be purchased pre-cleaned according to USEPA Office of Solid Waste and Emergency Response (OSWER) Directive 9240.05A requirements. Certificates of analysis will be filed in the project file.

11.2.2 Field Duplicates

Field duplicates will be collected from the different site materials to verify the reproducibility of the sampling methods. Field duplicates will be prepared by placing aliquots from the same sample location into individual sample containers, which are submitted blind to the laboratory. In general, field duplicates will be analyzed at a 5 percent frequency (every 20 samples) for the chemical constituents. Table 1 provides an estimated number of field duplicates to be prepared for each applicable parameter and matrix.

11.2.3 Rinse Blanks

Rinse blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Rinse blanks will be prepared and submitted for analysis once per day per matrix. Rinse blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) which has been routed through a cleaned sampling device. When dedicated sampling devices are used or sample containers are used to collect the samples, rinse blanks will not be necessary. Table 1 provides an estimated number of rinse blanks for environmental media samples to be collected during the removal action.

11.3 Analytical Laboratory Quality Control Checks

11.3.1 General

Internal laboratory quality control checks will be used to monitor data integrity. These checks will include method blanks, matrix spikes (and matrix spike duplicates), spike blanks, internal standards, surrogate samples, calibration standards, and reference standards. Project QC limits for duplicates and matrix spikes are identified in Table 2. Laboratory control charts will be used to determine long-term instrument trends.

11.3.2 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, need to be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank is initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources which could affect sample analysis. One method blank will be analyzed with each analytical series associated with no more than 20 samples.

11.3.3 Matrix Spikes/Matrix Spike Duplicates

Matrix spikes and matrix spike duplicates will be used to measure the accuracy of analyte recovery from the sample matrices. Matrix spikes and matrix spike duplicates will be site-specific. Matrix spike duplicate pairs will be analyzed at a 10 percent frequency (every 10 samples or once every week, whichever comes first).

When matrix spike recoveries are outside QC limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and determine the effect on the reported sample results. Table 1 presents an estimated number of matrix spike and matrix spike duplicate analyses for each applicable matrix and parameter.

11.3.4 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding instrument stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours, or more frequently as specified in the applicable analytical method. If results of the calibration check standard exceed specified tolerances, then samples analyzed since the last acceptable calibration check standard will be reanalyzed.

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods as summarized in Section 13.

11.3.5 Reference Standards/Control Samples

Reference standards are standards of known concentration, and independent in origin from the calibration standards. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes the preparation of calibration standards, the validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods.

11.4 Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision will be monitored through the use of matrix spike/matrix spike duplicate sample analyses.

The precision of data will be measured by calculation of the relative percent difference (RPD) by the following equation:

$$RPD = \frac{(A-B)}{(A+B)/2} \times 100$$

Where:

A = Analytical result from one of two duplicate measurements

B = Analytical result from the second measurement

Precision objectives for duplicate analyses are identified in Table 2.

11.5 Data Accuracy Assessment Procedures

Laboratory accuracy will be assessed via the use of matrix spikes, surrogate spikes and reference standards. Where available and appropriate, QA performance standards will be analyzed periodically to assess laboratory accuracy. Accuracy will be calculated in terms of percent recovery as follows:

$$\%\ \text{Recovery} = \frac{A-X}{B} \times 100$$

Where:

A = Value measured in spiked sample or standard

X = Value measured in original sample

B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy between the original and spiked measurements. Accuracy objectives for matrix spike recoveries are identified in Table 2.

11.6 Data Completeness Assessment Procedures

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

$$\text{Completeness} = \frac{\text{Number valid results}}{\text{Total number of results generated}} \times 100$$

As a general guideline, overall project completeness is expected to be at least 80 percent. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

12. Instrument/Equipment Testing, Inspection and Maintenance Requirements

12.1 General

Laboratory instrument and equipment documentation procedures are provided in Attachment B. Documentation includes details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (which will include information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. Tables of preventive maintenance procedures for instruments are included in Attachment B. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

12.1.1 Instrument Maintenance

Maintenance schedules for laboratory equipment adhere to the manufacturer's recommendations. Tables of preventive maintenance procedures for instruments are included in Attachment B. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with manufacturer or qualified contractors. Paperwork associated with service calls and preventative maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventative maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used, and/or the judgment of the Laboratory Systems Manager.

All major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.

13. Instrument Calibration and Frequency

13.1 Laboratory Instrument and Equipment

When analyses are conducted according to the USEPA SW-846 methods, the calibration procedures and frequencies specified in the applicable method will be followed. For analyses governed by SOPs, see the appropriate SOP in Attachment B for the required calibration procedures and frequencies. Records of calibrations will be filed and maintained by the laboratory. These records will be subject to QA audit. For all instruments, the laboratory will maintain trained repair staff with in-house spare parts or will maintain service contracts with vendors.

All standards used in the calibration of equipment are traceable, directly or indirectly, to National Institute of Standards and Technology (NIST). All standards received are logged into standard receipt logs maintained by the individual analytical groups. Each group maintains a standards log which tracks the preparation of standards used for calibration and QC purposes.

Total Mercury (Method 7471)

Atomic absorption instruments are calibrated using a minimum of five standards and a blank. The initial calibration is verified prior to the analysis of samples by an initial calibration verification standard (ICV). The recovery of this standard must be between 90 and 110 percent for the initial calibration to be considered valid.

Continuing calibration verification (CCV) standards are analyzed every 10 samples. The recovery of this standard must be between 80 and 120 percent. In addition, a final CCV must be analyzed at the end of the analytical sequence. Recovery of this standard must also be between 80 and 120 percent. If any of the CCVs (including the final CCV) fail to meet method specifications, all samples analyzed since the last compliant standard must be reanalyzed for the failed analyte(s).

Methyl Mercury (Method 1630 – Modified for Sediment Matrix)

The Cold Vapor Atomic Florescence Spectrometer (CVAFS) is calibrated by analysis of standard solutions at five concentration levels as well as an ethylation blank. The standard responses are corrected for blank content and the calibration factors (CFs) are determined using a response versus concentration calculation. If the percent relative standard deviation (% RSD) for the calibration factors is less than 15% and if the recovery of the lowest standard is between 65 and 135%, the curve is considered valid and analysis may begin.

CCV standards are run every 12 hours, or every 20 samples, whichever is more frequent. If the recovery of the CCV is within 77 and 123%, the continuing calibration is considered valid and analysis may continue. A CCV must be analyzed at the end of the analytical sequence. The recovery of this standard must also be within 77 and 123%. If any of CCV (including the final CCV) fail to meet method specifications, all samples analyzed since the last compliant standard must be reanalyzed.

14. Inspection/Acceptance Requirements for Supplies and Consumables

Inspection/acceptance requirements for supplies and consumables are as specified in Attachment B.

15. Data Acquisition Requirements for Nondirect Measurements

Prior to their use, historic data sets will be reviewed according to the procedures identified in subsequent sections of this QA/SAPP to determine the appropriate uses of such data. The extent to which these data can be validated will be determined by the analytical level and QC data available. The evaluation of historic data for SRA purposes requires the following:

- Identification of analytical levels;
- Evaluation of QC data, when available; and
- Development of conclusions regarding the acceptability of the data for intended uses.

Acceptability of historic data for intended uses will be determined by application of these procedures and professional judgment. If the historic data quality cannot be determined, its use will be limited to general trend evaluations.

16. Data Management

The purpose of the data management is to ensure that all of the necessary data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The field investigations will encompass a large number of samples and a variety of sample matrices and analytes from a large geographic area. From the large amount of resulting data, the need arises for a structured, comprehensive, and efficient program for management of data. An overview of the data management process is provided in Figure 4.

As outlined in the SRAMP, sampling activities will include analyses for total and methyl mercury. The data management program established for the project includes field documentation and sample QA/QC procedures, methods for tracking and managing the data, and a system for filing all site-related information. More specifically, data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and accurate. These procedures are described in detail in the following section.

The data management plan has five elements: 1) sample designation system, 2) field activities, 3) sample tracking and management, 4) data management system,an d 5) document control and inventory.

16.1 Sample Designation System

A concise and easily understandable sample designation system is an important part of the project sampling activities. It provides a unique sample number that will facilitate both sample tracking and easy re-sampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sampled collected, as outlined in Section 6.1.1.

16.2 Field Activities

Field activities designed to gather the information necessary to make decisions regarding the off-site areas require consistent documentation and accurate record keeping. During site activities, standardized procedures will be used for documentation of field activities, data security, and QA. These procedures are described in further detail in the following subsections.

16.2.1 Field Documentation

Complete and accurate record keeping is a critical component of the field investigation activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and validation process. To ensure that the field investigation is thoroughly documented, several different information records, each with its own specific reporting requirements, will be maintained, including:

- Field logs; and
- Chain-of-custody forms.

A description of each of these types of field documentation is provided below.

Field Logs

The personnel performing the field activities will keep field logs that detail all observations and measurements made during the remedial investigation. Data will be recorded directly into site-dedicated, bound notebooks, with each entry dated and signed. To ensure at any future date that notebook pages are not missing, each page will be sequentially numbered. Erroneous entries will be corrected by crossing out the original entry, initialing it, and then documenting the proper information. In addition, certain media sampling locations will be surveyed to accurately record their locations. The survey crew will use their own field logs and will supply the sampling location coordinates to the File Custodian.

Chain-of-Custody Forms

COC forms are used as a means of documenting and tracking sample possession from time of collection to the time of disposal. A COC form will accompany each field sample collected, and one copy of the form will be filed in the field office. All field personnel will be briefed on the proper use of the COC procedure. A more thorough description of the COC forms is located in the SOPs (Appendix B).

16.2.2 Data Security

Measures will be taken during the field investigation to ensure that samples and records are not lost, damaged, or altered. When not in use, all field notebooks will be stored at the field office or locked in the field vehicle overnight. Access to these files will be limited to the field personnel who utilize them.

16.3 Sample Management and Tracking

A record of all field documentation, as well as analytical and QA/QC results, will be maintained to ensure the validity of data used in the site analysis. To effectively execute such documentation, carefully constructed sample tracking and data management procedures will be used throughout the sampling program.
Sample tracking will begin with the completion of COC forms, as described in Appendix B and summarized in Section 9.2.3. On a daily basis, the completed COC forms associated with samples collected that day will be faxed from the project office to the QAR. Copies of all completed COC forms will be maintained in the field office. On the following day, the QAR will telephone the laboratory to verify receipt of samples.

When analytical data are received from the laboratories, the QAR will review the incoming analytical data packages against the information on the COCs to confirm that the correct analyses were performed for each sample and that results for all samples submitted for analysis were received. Any discrepancies noted will be promptly followed-up by the QAR.

16.4 Data Management System

In addition to the sample tracking system, a data management system will be implemented. The central focus of the data management system will be the development of a personal computer-based project database. The project database, to be maintained by the Database Administrator, will combine pertinent geographical, field, and analytical data. Information that will be used to populate the database will be derived from three primary sources: surveying of sampling locations, field observations, and analytical results. Each of these sources is discussed in the following sections.

16.4.1 Computer Hardware

The database will be constructed on Pentium®-based personal computer work stations connected through a Novell network server. The Novell network will provide access to various hardware peripherals, such as laser printers, backup storage devices, image scanners, modems, etc. Computer hardware will be upgraded to industrial and corporate standards, as necessary, in the future.

16.4.2 Computer Software

The database will be written in Microsoft Access, running in a Windows operating system. Custom applets, such as diskette importing programs, will be written in either Microsoft VBA or Microsoft Visual Basic.

Geographic Information System (GIS) applications will be developed in ERSI ArcView, with additional customization performed with Visual Basic. Tables and other database reports will be generated through Access in conjunction with Microsoft Excel, Microsoft Word, and or Seagate Crystal Reports. These software products will be upgraded to current industrial standards, as necessary.

16.4.3 Survey Information

In general, each location sampled as part of the SRA will be surveyed to ensure accurate documentation of sample locations for mapping and GIS purposes, to facilitate the re-sampling of select sample locations during future monitoring programs, if needed, and for any additional activities. The surveying activities that will occur in the field will consist of the collection of information that will be used to compute a northing and easting in state plane coordinates for each sample location and the collection of information to compute elevations relative to the National Geodetic Vertical Datum of 1988 for select sample locations, as appropriate. All field books associated with the surveying activities will be stored as a record of the project activities.

Conventional surveying techniques will be used to gather information such as the angle and distance between the sample location and the control monument, as well as point attributes. This information will be digitally stored in a data logger attached to the total station. On a regular basis, each data logger in use will be transferred to the BBL Syracuse, New York office, where the information will be downloaded into a personal computer for processing with surveying software. Control monuments will be established using GPS techniques. The surveying software allows the rapid computation of a location's state plane coordinates.

Differential leveling techniques will be used to gather information to be used to compute a sample location's elevation. During the differential leveling process, which includes at least one benchmark of known elevation, detailed field notes will be kept in a field book. On a weekly basis, a copy of the relevant pages will be forwarded to the Syracuse, New York office, where the relevant information will be manually keyed into BBL's surveying software package for further processing. The surveying software reduces the field notes and calculates a location's elevation relative to the project datum.

Following computation of a location's state plane coordinates and, at select locations, elevations, the computer information will undergo a QA/QC review by a licensed land surveyor. Following the approval of the computed information, the coordinates and elevations will be transferred to the File Custodian both in a digital and a hard copy format. This data will then be loaded into the database and linked to the field and analytical data.

16.4.4 Field Observations

An important part of the information that will ultimately reside in the data management system for use during the project will originate in the observations that are recorded in the field. Figure 4 depicts the general data flow of the field observations from sample collection through inclusion in the data management system.

Following each sampling event, a status memorandum will be prepared by the field personnel who performed the sampling activities. The purpose of the status memo is to present a summary and a record of the sampling event. Topics to be discussed include the locations sampled, the sampling methodologies used, QA/QC procedures, blind duplicate and MS/MSD sample identification numbers, equipment decontamination procedures, personnel involved in the activity, and any other noteworthy events that occurred.

Status memos are tools used to keep project personnel informed on the details of the field activities and are also invaluable during the development of the final report. Each status memo will be reviewed for accuracy and completeness by the respective sampling activity manager. Following the approval and finalization of each memo, the status memo will be used to transfer field observations into the data management system.

16.4.5 Analytical Results

Analytical results will be provided by the laboratories in both a digital and a hard copy format. Upon receipt of each analytical package, the original COC form will be placed in the project files. The data packages will be examined to ensure that the correct analyses were performed for each sample submitted and that all of the analyses requested on the COC form were performed. If discrepancies are noted, the QAR will be notified and will promptly follow up with the laboratory to resolve any issues.

Each data package will be validated in accordance with the procedures presented in Section 20.1. Any data that does not meet the specified standards will be flagged pending resolution of the issue. The flag will not be removed from the data until the issue associated with the sample results is resolved. Although flags may remain for certain data, the use of that data may not necessarily be restricted.

Following completion of the data validation, the digital files will be used to populate the appropriate database tables. An example of the format of electronic data deliverable (EDD) format is included in Table 5. This format specifies one data record for each constituent for each sample analyzed. Specific fields include:

- Sample identification number;

- Date sampled;
- Date analyzed;
- Parameter name;
- Analytical result;
- Units;
- Detection limit; and
- Qualifier(s).

The individual EDDs, supplied by the laboratory in either an ASCII comma separated value (CSV) format or in a Microsoft Excel worksheet, will be loaded into the appropriate database table via a custom-designed user interface Visual Basic program. Any analytical data that cannot be provided by the laboratory in electronic format will be entered manually. After entry into the database, the EDD data will be compared to the field information previously entered into the database to confirm that all requested analytical data have been received.

16.4.6 Data Analysis and Reporting

The database management system will have several functions to facilitate the review and analysis of the SRA data. Routines have been developed to permit the user to scan analytical data from a given site for a given media. Several output functions are also available which can be modified, as necessary, for use in the data management system.

A valuable function of the data management system will be the generation of tables of analytical results from the project databases. The capability of the data management system to directly produce tables reduces the redundant manual entry of analytical results during report preparation and precludes transcription errors that may occur otherwise. This data management system function creates a digital file of analytical results and qualifiers for a given media. The file can then processed into a table of rows and columns which can be transferred to word processing software (e.g., Microsoft Word) for final formatting and addition of titles and notes. Tables of analytical data will be produced as part of data interpretation tasks and the reporting of data to USEPA.

Another function of the data management system will be to create digital files of analytical results and qualifiers suitable for transfer to mapping presentation software. A function has been created by BBL that creates a digital file consisting of sample location number, state plane coordinates, sampling date, and detected constituents and associated concentrations and analytical qualifiers. The file is then transferred to an AutoCAD work station,

17. Assessment and Response Actions

17.1 General

Performance and systems audits will be completed in the field and laboratory during the SRA as described below.

17.2 Field Audits

The following field performance and systems audits will be completed during this project.

The appropriate Task Manager will monitor field performance. Field performance audit summaries will contain an evaluation of field measurements to verify that measurements are taken according to established protocols. The BBL QAR will review field reports and communicate concerns to the BBL Remedial Project Manager and/or Task Managers, as appropriate. In addition, the BBL QAR will review the rinse and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this document with actual QA/QC activities completed will be performed. The appropriate Task Manager and QAR will periodically confirm that work is being performed consistent with this QA/SAPP and the SRAMP. USEPA may also conduct Field Audits in keeping with customary logistic arrangements with Solutia, Inc.

17.3 Laboratory Audits

Annual internal laboratory audits are conducted by each analytical laboratory's QAR. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratories' Quality Assurance Manuals and SOPs. The results of the audits are summarized and issued to each department supervisor, the laboratory manager, the laboratory director, and the corporate QA director. A systems audit of each laboratory is performed annually by the QARs to determine if the procedures implemented by each laboratory are in compliance with the corporate quality assurance plan.

In addition to the laboratory's internal audits and participation in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

BBL reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the course of the project, as deemed necessary. USEPA may also conduct Laboratory Audits in keeping with customary logistic arrangements with the laboratory and Solutia, Inc.

17.4 Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QA/SAPP or the SRAMP. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. In the event that critical data of sufficient quality are not available in adequate quantity to meet project objectives, re-sampling will be conducted as necessary. Field and laboratory corrective action procedures for the actions are described below.

17.4.1 Field Procedures

When conducting the action field work, if a condition is noted that would have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented will be documented on a Corrective Action Form and reported to the appropriate BBL Task Manager, QAR, and Remedial Project Manager.

Examples of situations that would require corrective actions are provided below:

- Protocols as defined by the QA/SAPP and SRAMP have not been followed;
- Equipment is not in proper working order;
- QC requirements have not been met; or
- Issues resulting from performance or systems audits have not been resolved.

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

17.4.2 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse effect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action to be taken will be documented and reported to the appropriate Remedial Project Manager and QAR.

Corrective action may be initiated, at a minimum, under the following conditions:

- Protocols as defined by this QA/SAPP have not been followed;
- Predetermined data acceptance standards are not obtained;
- Equipment is not in proper working order or calibrated;
- Sample and test results are not completely traceable;
- QC requirements have not been met; or
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. For all instrument systems in use at Battelle and Brooks Rand, corrective action is initiated at a point were the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control) it is brought to the attention of the QAR and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits, the analytical results of split samples, or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilutions, matrix modifications, re-sampling, or other activities.

18. Reports to Management

The QAR will audit the implementation of the QA/SAPP. Each project component will result in some type of QA report or, by its absence, acknowledge that no significant QA or QC deviations occurred. Items that may result in a QA report include:

- Changes or updates to the QA/SAPP;
- Deviations from QA/SAPP or SRAMP specification;
- The results of system and performance audits;
- Significant QA/QC problems, recommended solutions, and the results of corrective actions; and
- Limitations on the use of measurement data.

QA reports will be generated by each analytical laboratory and submitted to BBL with the final data package. BBL will include QA statements in its report to Solutia, Inc., as well the final report submitted to the USEPA.

18.1 Field Reports

Reporting of the quality of field sample collection and field measurements will be the responsibility of the Field Supervisor or designee. Information from the field logbooks will be compiled and a summary report on field activity QA will be prepared for the project file.

18.2 Laboratory Reports

The laboratory will maintain QA records related to analyses, quality control, and corrective action. This information will be made available to the Remedial Project Manager upon request. Routine reporting will include documenting of all internal quality control checks performed for this project.

19. Data Review, Validation and Verification

19.1 General

After field and laboratory data are obtained, the data will be subject to the following:

1. Reduction, or manipulation mathematically, or otherwise into meaningful and useful forms;
2. Review;
3. Organization, interpretation, and reporting; and
4. Data validation.

19.2 Field Data Reduction and Review

19.2.1 Field Data Reduction

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets, and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the SRAMP and this QA/SAPP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

19.2.2 Field Data Review

Field data calculations, transfers, and interpretations will be conducted by the field personnel and reviewed for accuracy by the appropriate Task Manager and the QAR. Logs and documents will be checked for:

1. General completeness;
2. Readability;
3. Usage of appropriate procedures;
4. Reasonableness in comparison to present and past data collected;
5. Correct sample locations; and
6. Correct calculations and interpretations.

19.3 Laboratory Data Reduction and Review

19.3.1 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data will be entered into permanently bound laboratory notebooks. The data entered are sufficient to document all factors used to arrive at the reported value.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation will be performed using either internal or external standards.

Inorganic analyses will be based on regression analysis. Regression analysis is used to fit a curve through the calibration standard data. The sample concentrations will be calculated using the resulting regression equations. Non-aqueous values will be reported on a dry-weight basis. Unless otherwise specified, all values will be reported uncorrected for blank contamination.

19.3.2 Laboratory Data Review

Data will be subject to multi-level review by each analytical laboratory. The group leader will review data reports prior to release for final data report generation. The QAR will review all of the final data reports, and the laboratory director will review a cross section of the final data reports. Final data reports are reviewed by the Department Manager prior to shipment to BBL.

If discrepancies or deficiencies exist in the analytical results, then corrective action will be taken, as discussed in Section 17. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form. This form will be submitted to the BBL Remedial Project Manager.

19.4 Data Validation and Verification

All data generated will be subjected to the data validation and verification procedures outlined in Section 20.

20. Validation and Verification Methods

20.1 Data Validation and Verification

Data validation entails a review of the QC data and the raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs, and which, if any, environmental samples were related to any out-of-control QC samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

BBL will prepare an SOP for validating the total and methyl mercury analytical data using the most recent versions of the USEPA's Function Guidelines (USEPA, 1999, 2002) as a model, where appropriate[1]. These procedures and criteria may be modified as necessary to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the data quality objectives. Data validation will include a review of completeness and compliance, including, but not limited to, the elements provided in Table 6.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QA/SAPP. Any deviations from the analytical method or any special reporting requirements apart from that specified in this QA/SAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package;
- Verify that field COC forms were completed and that samples were handled properly;
- Verify that holding times were met for each parameter. Holding time exceedances, should they occur, will be documented. Data for all samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis;
- Verify that parameters were analyzed according to the methods specified;

[1] This SOP was discussed with USEPA Region 5 QA representative Mr. R. Byvik on 22 January 2003. The SOP will be submitted for USEPA review and approval prior to beginning sample analysis.

- Review QA/QC data (i.e., make sure duplicates, blanks, and spikes were analyzed on the required number of samples, as specified in the method; verify that duplicate and matrix spike recoveries are acceptable);
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Remedial Project Manager and/or laboratory manager, as appropriate; and
- If data appears suspect, investigate the specific data of concern. Calculations will be traced back to raw data; if calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in the form of a written QA report addressing the following topics as applicable to each method:

- Assessment of the data package;
- Description of any protocol deviations;
- Failures to reconcile reported and/or raw data;
- Assessment of any compromised data;
- Laboratory case narrative;
- Overall appraisal of the analytical data; and
- Table of site name, sample quantities, matrix, and fractions analyzed.

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without QC qualifiers. Qualified data can provide useful information.

Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for reanalysis may be made by the BBL QAR at this point.

Data validation reports will be kept in the project file at the BBL office in Syracuse, New York.

21. Reconciliation with User Requirements

The data results will be examined to determine the performance that was achieved for each data usability criteria. The performance will then be compared with the project objectives. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Action options may include any or all of the following:

- Retrieval of missing information;
- Request for additional explanation or clarification;
- Reanalysis of sample from extract (when appropriate); and
- Recalculation or reinterpretation of results by the laboratory.

These actions may improve the data quality, reduce uncertainty, and may eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following actions may be taken:

- Extrapolation of missing data from existing data points;
- Use of historical data; and
- Evaluation of the critical/non-critical nature of the sample.

If the data gap can not be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be performed. If the resultant uncertainty level is unacceptable, then the following action must be taken:

- Additional sample collection and analysis.

Acronyms and Abbreviations

ASTM	American Society for Testing and Material
BBL	Blasland, Bouck & Lee, Inc.
CCV	Continuing Calibration Verification
CFs	Calibration Factors
CLP	Contract Laboratory Program
COC	Chain-of-Custody
CSV	Comma Separated Value
CVAFS	Cold Vapor Atomic Fluorescence Spectrometer
DGPS	Differential Global Positioning System
DUP	Duplicate
DQOs	Data Quality Objectives
EDD	Electronic Data Deliverable
GIS	Geographic Information System
ICB	Initial Calibration Blank
ICV	Initial Calibration Verification
mg/kg	Milligrams per kilogram
mS/cm	Millisiemens per centimeter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NBS	Nations Bureau of Standards
NEIC	National Enforcement Investigations Center
NIST	National Institute of Science and Technology
NPL	National Priorities List
OSHA	Occupational Safety and Health Administration
OSWER	Office of Solid Waste and Emergency Response
PID	Photoionization Detector
PPE	Personal Protective Equipment
ppb	Parts per billion
ppm	Parts per million
QAR	Quality Assurance Reviewer
QA/SAPP	Quality Assurance/Sampling and Analysis Project Plan
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation Recovery Act
RSD	Relative Standard Deviation
SDG	Sample Delivery Group
Solutia	Solutia Inc.
SOP	Standard Operating Procedures
SRA	Sediment Removal Action
SRAMP	Sediment Removal Action Mitigation Plan
TSCA	Toxic Substances Control Act
TSP	Trisodium phosphate
SU	Standard Units
USCS	Unified Soil Classification System
USGS	United States Geological Survey
USEPA	United States Environmental Protection Agency

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Tables

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engineers & scientists

Table 1

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Environmental and Quality Control Analyses

Parameter	Estimated Environmental Sample Quality	Field QC Analyses						Laboratory QC Sample						Total
		Trip Blank		Rinse Blank		Field Duplicate		Matrix Spike		Matrix Spike Duplicate		Lab Duplicate		
		Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	Freq.	No.	
Surface Sediment														
Total Mercury	60	NA	--	1/day	7	1/20	3	2/20	6	2/20	6	NA	--	76
Methyl Mercury	60	NA	--	1/day	7	1/20	3	2/20	6	2/20	6	NA	--	76
Subsurface Sediment														
Total Mercury	30	NA	--	1/day	7	1/20	2	2/20	3	2/20	3	NA	--	43
Methyl Mercury	30	NA	--	1/day	7	1/20	2	2/20	3	2/20	3	NA	--	43

Notes:
1/day One rinse blank per day or one per 20 samples, whichever is more frequent.
Freq Frequency
NA Not Applicable
No. Number
QC Quality Control

Table 2

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Analytical Quality Control Limits[a]

Parameter	Field Duplicates RPD	Accuracy - % Recovery		Precision - RPD	
		MS/MSD	Surrogate	MS/MSD	Duplicate[b]
Sediment					
Total Mercury	100	80-120	--	30	--
Methyl Mercury	100	65-135	--	35	--

Notes:

[a] The listed QC limits are based on USEPA guidance and are advisory. However, frequent failures to meet the QC limits warrant investigation of the laboratory

[b] Duplicate control limits apply to laboratory duplicates.

MS Matrix Spike
MSD Matrix Spike Duplicate
RPD Relative Percent Difference

Table 3

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Parameters, Methods and Target Reporting Limits

Analyte	Method[a]	Reporting Limit		
		Sediment	Water	Biota
Total Mercury	7471[a]	0.02 mg/kg	--	--
Methyl Mercury	1630[b]	0.0394 ug/kg	--	--

Notes:

[a] USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste* SW-846 3rd ed. Washington, D.C. 1996.

[b] USEPA. Office of Science and Technology. *Methyl Mercury in Water by Distillation, Aqueous Ethylation, Pugeal Trap, and CVAFS.* EPA-821-R-01-020. 2001

Table 4

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Sample Containers, Preservation, and Holding Times

Parameter	Method[a]	Bottle Type	Preservation	Holding Time[b]
Sediment				
Total Mercury	SW-846-7471	250 ml plastic or glass jar	Cool to 4°C	28 days to analysis
Methyl Mercury	USEPA 1630	125 ml borosilicate glass jar with Teflon©-lined lid; minimize headspace	Cool to 4°C minimize headspace	48 hours

Notes:

[a] USEPA. Office of Solid Waste and Emergency Response. *Test Methods for Evaluating Solid Waste.* SW-846 3rd ed. Washington, D.C. 1996. and

USEPA. Office of Science and Technology. *Methyl Mercury in Water by Distillation. Aqueous Ethylation, Purge and Trap, and CVAFS.* EPA-821-R-01-020. 2001

[b] All holding times are measured from date of collection.

Table 5

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Electronic Data Deliverable Format

Field Name	Maximum Length	Data Type	Comments
SAMPLEID	20	TEXT	
SDG	20	TEXT	
LABSAMPLEID	30	TEXT	
QAQTYPE	40	TEXT	FIELD DUP, DILUTION, REANALYSIS, TRIP BLANK, etc.
LABMETHOD	50	TEXT	
PARAMETER	100	TEXT	
SAMPLEDATE	10	DATE	MM/DD/YY
EXTRACTDATE	10	DATE	MM/DD/YY
ANALYSISDATE	10	DATE	MM/DD/YY
CASNO	40	TEXT	
RESULTS	20	NUMERIC	Numeric Results ONLY
TEXTRESULTS	100	TEXT	Used for non-numeric results
DILUTION	20	NUMERIC	Dilution factor
DETECTLIMIT	20	NUMERIC	
CONCUNITS	20	TEXT	
LABQUALIFIER	10	TEXT	
VALQUALFIER	10	TEXT	
COMMENTS	256	TEXT	

Notes:

This definition is for comma separated value (CSV) format. Text data must be in double quotation marks. Numeric data and dates do not need double quotes.

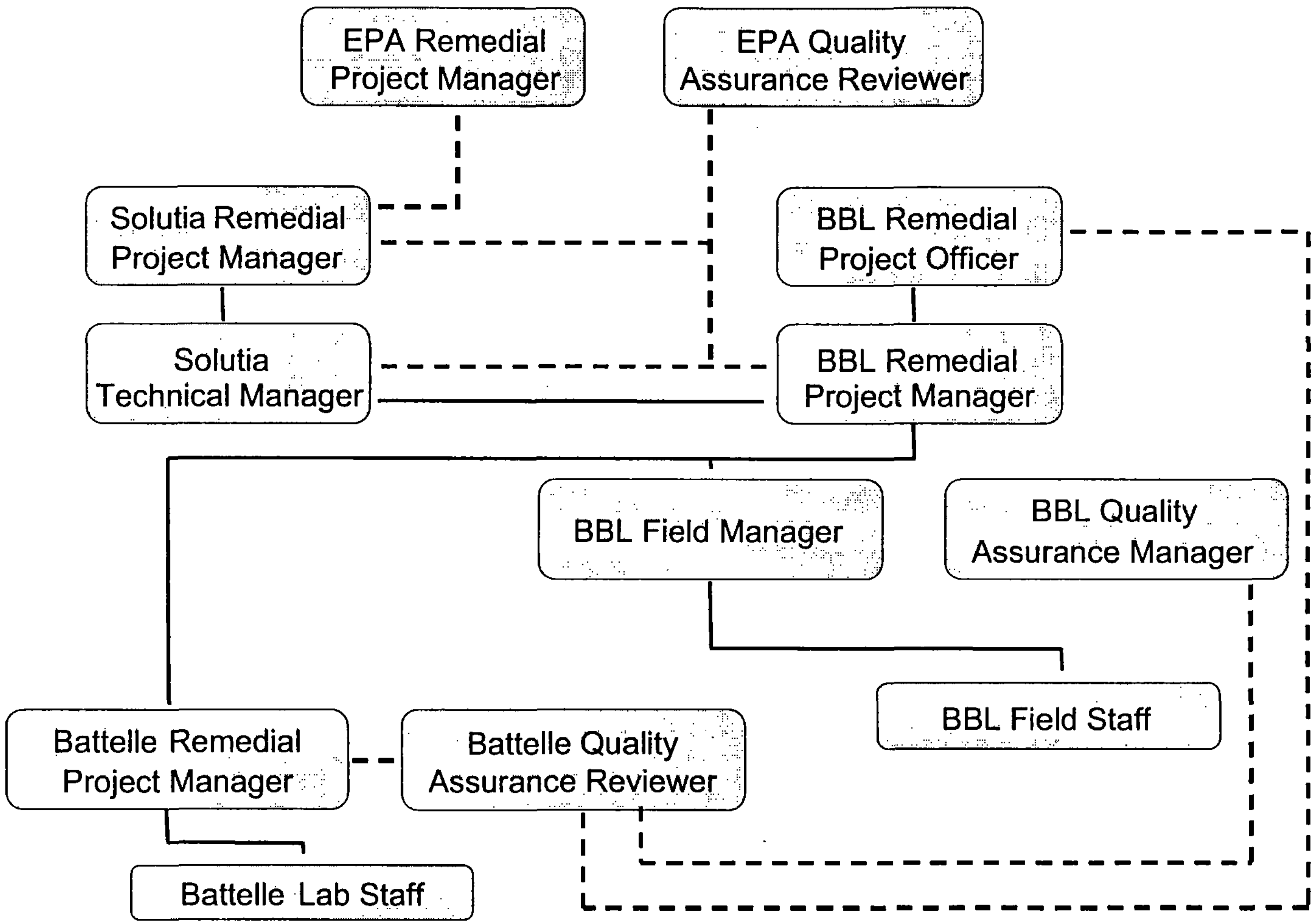
Table 6

Solutia Inc.
Sauget Illinois
Sauget Area 1 - Dead Creek Sediment Removal Action

Data Validation Checklist

Review for Completeness	
1.	All chain-of-custody forms included.
2.	Case narratives.
3.	QA/QC summaries of analytical data including supporting documentation.
4.	All relevant calibration data including supporting documentation.
5.	Instrument and method performance data.
6.	Documentation showing laboratory's ability to attain specified method detection limits.
7.	Data report forms of examples for calculations of concentrations.
8.	Raw data used in identification and quantification of the analysis required.
Review of Compliance	
1.	Data package completed as described above.
2.	QAPP requirements for data production and reporting have been met.
3.	QA/QC criteria have been met.
4.	Instrument type and calibration procedures have been met.
5.	Initial and continuing calibration have been met.
6.	Data reporting forms are completed.
7.	Problems and corrective actions documents.

Figures



SOLUTIA, INC.
SAUGET, ILLINOIS

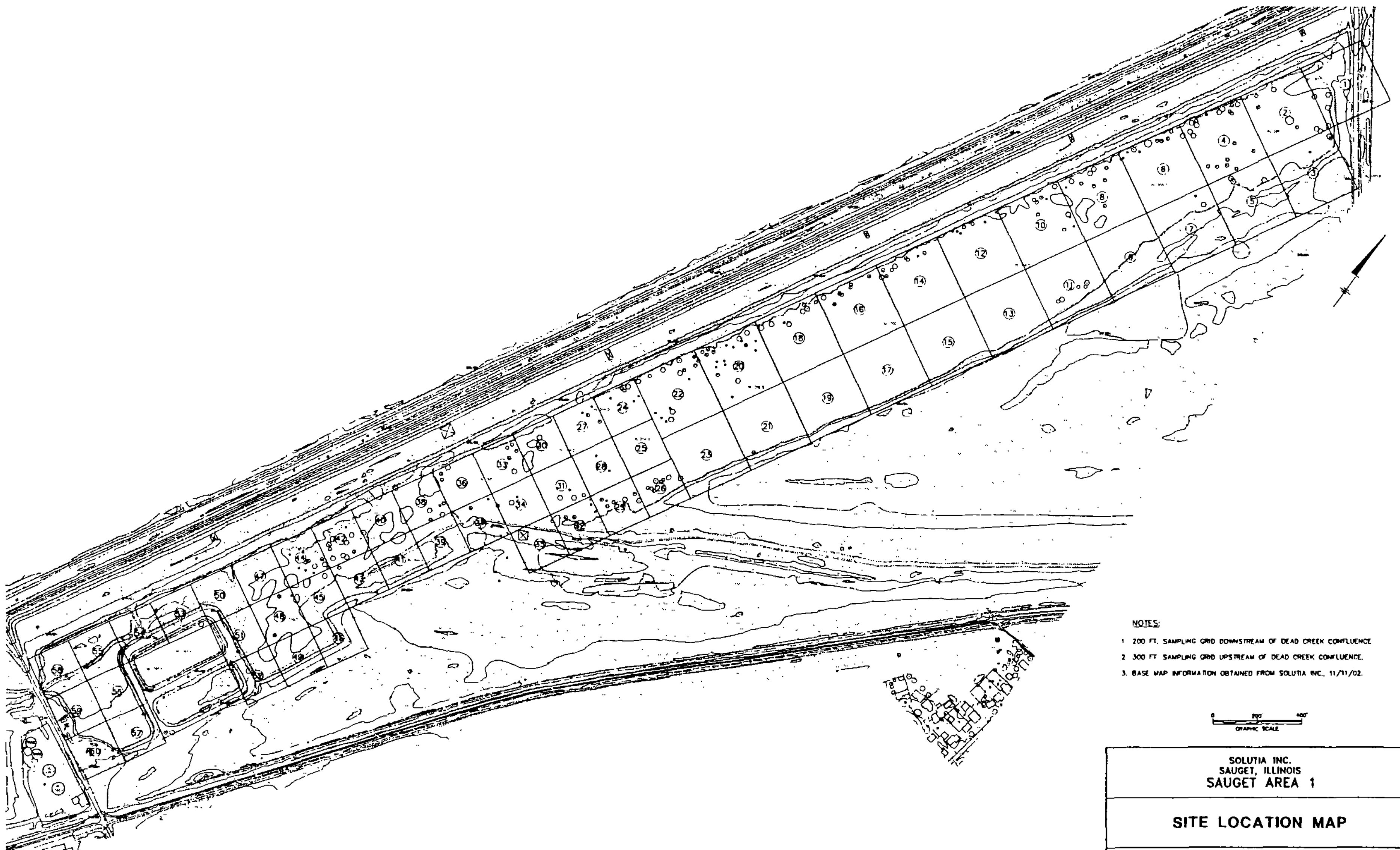
QA/SAPP

ORGANIZATION CHART

BBL BLASLAND, BOUCK & LEE, INC. *engineers & scientists*

FIGURE 1

ms_Figure 1.xls_Figure 1_5/1/2003



NOTES:

1. 200 FT. SAMPLING GRID DOWNSTREAM OF DEAD CREEK CONFLUENCE
2. 300 FT. SAMPLING GRID UPSTREAM OF DEAD CREEK CONFLUENCE.
3. BASE MAP INFORMATION OBTAINED FROM SOLUTIA INC., 11/11/02.

0 200' 400'
GRAPHIC SCALE

SOLUTIA INC.
SAUGET, ILLINOIS
SAUGET AREA 1

SITE LOCATION MAP

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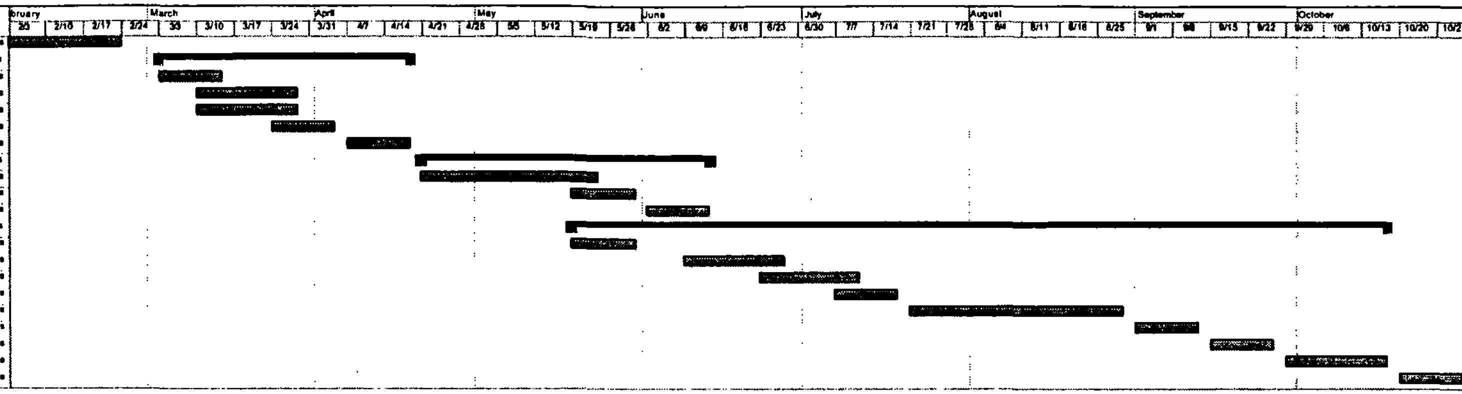
engineers & scientists

FIGURE 2

X: 10294X01.DWG
L: ON=*, OFF=REF*
P: PAGESET/PLT-DL
12/05/02 SYR-54-DJP LJP KMD
10294001/10294001.DWG

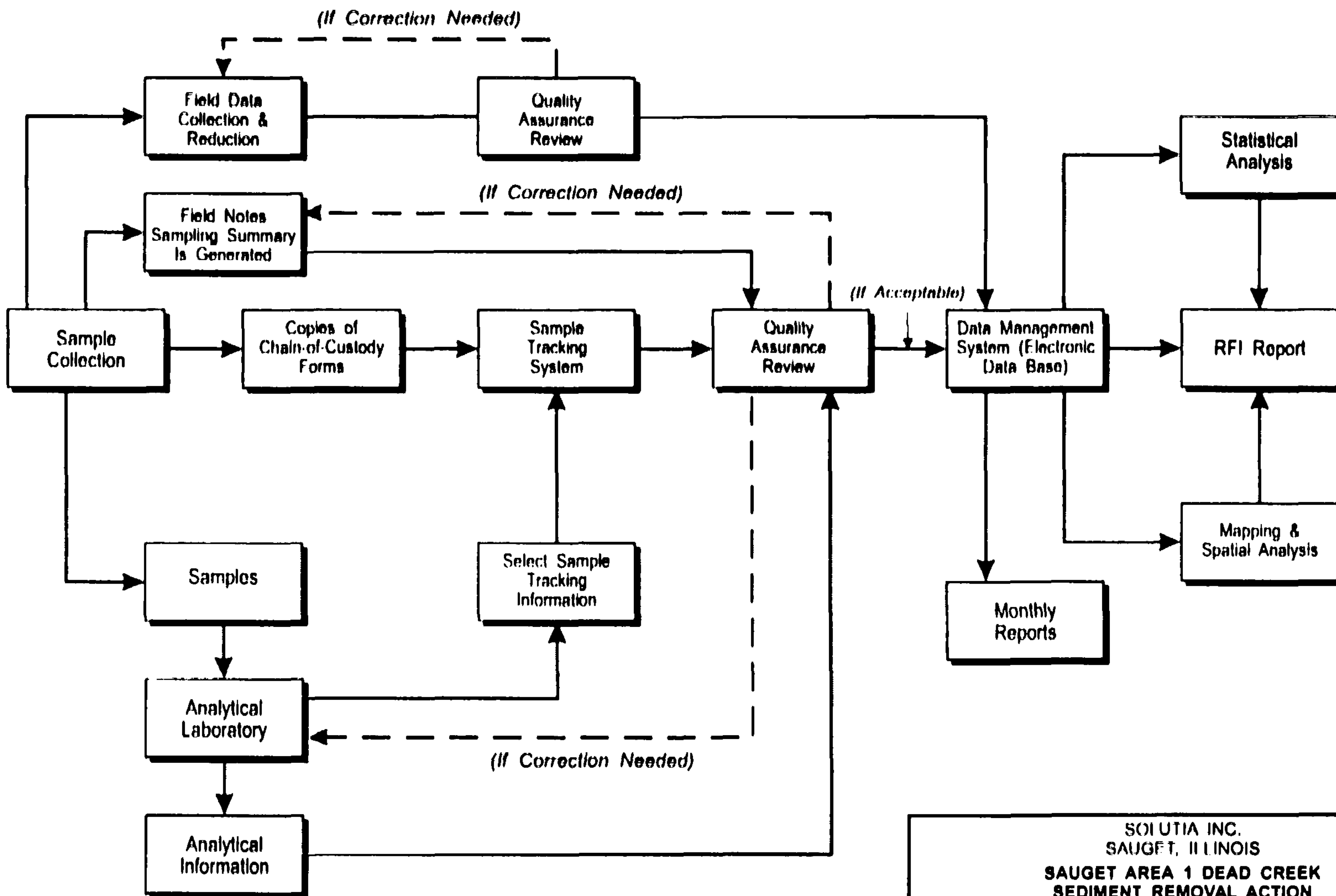
FIGURE 3. PROJECT TIMELINE

ID	Task Name	Duration
1	EPA APPROVAL OF QAPP	15 days
2	FIELD WORK	35 days
3	Sample Location Survey	10 days
4	Sediment Characterization	15 days
5	Sediment Sampling	15 days
6	Field Reporting	10 days
7	Data Review and Validation	10 days
8	LABORATORY ANALYSIS	40 days
9	Laboratory Analysis	25 days
10	Data Review and Validation	10 days
11	Data Evaluation	10 days
12	REPORTING	110 days
13	Data Validation Report	10 days
14	Draft Sediment Report Preparation	15 days
15	Internal Review of Draft Report	15 days
16	Submittal of Draft Report to EPA	10 days
17	EPA Review of Draft Report	30 days
18	Draft Report Revisions	10 days
19	Draft Final Re-Submittal to EPA	10 days
20	EPA Review of Draft Final	15 days
21	EPA APPROVAL OF REPORT	10 days



Project: Project Timeline
Date: Thu 5/1/03

Task | Progress | Summary | External Tasks | Deadline
Split | Milestone | Project Summary | External Milestone



SOLUTIA INC.
SAUGET, ILLINOIS

SAUGET AREA 1 DEAD CREEK SEDIMENT REMOVAL ACTION

DATA MANAGEMENT FLOW CHART

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FIGURE
4

11/08/02 SYR-D54-DJH
10285020/10285F01.CDR

Appendices

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Appendix A

Sediment Sampling Procedures

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APPENDIX A

Sediment Sampling Procedures

I. Introduction

The general procedures to be utilized in obtaining sediment samples from the Creek are outlined below. Lexan® tubing will be the primary method used to collected sediment cores, the tubing may be replaced with a calibration rod if just probing is being performed. If the Creek bed cannot be penetrated by the Lexan® tubing due to large cobble, boulders, or bedrock, an attempt will be made using a standard split-spoon sampler or a stainless steel bucket auger.

Following collection, the sediment is transferred to the appropriate sample containers. Samples to be analyzed for methyl mercury will be placed in sample containers without headspace.

II. Materials

- Personal protective equipment (as required by the Health and Safety Plan);
- Cleaning equipment (as required in Appendix C);
- Boat;
- Stainless steel tray;
- Duct tape;
- Lexan® tubing with end caps;
- Stainless steel sediment coring device;
- Stainless steel core driver;
- Calibrated rod for sediment depth measurement;
- Stainless steel spatula;
- T-handle;
- Extension rods;
- Hacksaw;
- Six-foot rule or survey rod;
- Stainless steel or plastic ruler;

- Stainless steel spoons;
- Camera;
- Transport container with ice;
- Appropriate sample containers and forms; and
- Field notebook.

III. *Procedures for Lexan® Tube Sampling*

1. Identify the proposed sampling location in the field notebook along with other appropriate information collected during sediment sampling activities.

2. Don personal protective equipment.

3. Position boat over the sampling location and record station position.

4. Assemble the coring device by inserting the stainless steel core in to the sampling tube.

5. Insert the "eggshell" check valve mechanism into the tip of the sampling tube with the convex surface positioned inside the core tubing.

6. Screw the coring point onto the tip of the sampling tube.

7. Screw the handle onto the upper end of the sampling tube and add extension rods as needed.

8. Lower the sampler into a perpendicular position on the sediment to be sampled. Measure the depth of water (if any is present).

9. Using the T-handle, push the sampler by hand until refusal. Measure the depth of sediment. If the procedure is being performed to determine sediment depth (probing), a calibrated rod may be used in place of the sampling tube.

10. Rotate the sampler to shear off the core at the bottom and retrieve the device.

11. Keeping the device upright, cap the bottom of the sampling tube, slide the core out of the sampling tube, and cap the top of the tube. Wipe the bottom end dry and seal with duct tape.

12. Measure the length of sediment recovered and evaluate the integrity of the core. If the core is not suitably intact, repeat the coring procedure adjacent to the location attempted.

13. Transport the core sample to shore.

14. While still keeping the core upright, use a handsaw to make a horizontal cut in the tube approximately one inch above the sediment.

15. Re-cap the cut end of the tube, seal the cap with duct tape and mark this end "TOP."

16. Wipe the tube dry.

17. Place a completed sample label on the tube.

18. Record the following sediment characteristics in the field logbook:

    - Texture
    - Color
    - Presence of debris
    - Presence of an oily sheen
    - Biological structures
    - Odor

19. Record the following information on both the tube and on the cap: 1) sample number, 2) sampling date, and 3) sampling time.

20. Place the core upright in a container with ice.

21. Repeat the above procedures until all core samples are collected for the day.

22. Each sediment core will be extruded from the Lexan® tubing onto a stainless steel tray. The core will be then be photographed. The cores will be sectioned into a 0 to 6 inch depth increment and a 6 inch to bottom increment. Both the 0 to 6 inch and 6 inch to bottom intervals will be transferred to appropriate sampling containers for the odd numbered grid number, only the 0 to 6 inch interval shall be retained for the even numbered intervals. Samples to be analyzed for methyl mercury will be placed in sampling containers with minimized headspace.

23. Core sections may be frozen to facilitate sectioning when the sediment is extremely loose.

24. Samples will be stored in coolers on ice until transfer to the laboratory (to be received within 48 hours of sample collection). All sample containers will be labeled with: 1) site name, 2) project number, 3) grid number, 4) sample interval, 5) date, 6) time of collection, and 7) names of sampling personnel. All appropriate information will also be entered in to the field logbook.

25. Fill out chain of custody and handle, pack and ship in accordance with the procedures in Appendix B.

IV. Procedure for Sediment Probing

The metal calibration rod will be used to probe sediment depths. From a boat at each location, the water depth to sediment will be measured by probing with a surveyor's rod. The sediment depth will then be measured by pushing a calibrated 5 8 inch galvanized hollow pipe into the sediment until refusal using reasonable human force. The depth of the penetrated sediment will be noted by subtracting the length of the rod above the water surface and the water depth at the point being probed from the length of the entire rod. Location, depth, and time of measurement will be noted in the field logbook.

V. Survey

A field survey control program will be conducted using standard instrument survey techniques to document the sediment sampling locations.

Appendix B

Field Sample Packing, Handling, and Shipping Procedures

BLASLAND, BOUCK & LEE, INC.
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Appendix B

Field Sample Packing, Handling, and Shipping Procedures

I. Handling

1. Fill in sample label with:

   - Sample type (sediment, soil, etc.);
   - Project number and site name;
   - Sample identification code and other sample identification information, if applicable;
   - Analysis required;
   - Date;
   - Time sampled;
   - Sample type (composite or discrete); and
   - Preservative added, if applicable.

2. Cover the label with clear packing tape to secure the label onto the container.

3. Check the caps on the sample containers to ensure that they are tightly sealed.

4. Wrap the sample container cap with clear packing tape to prevent it from becoming loose.

5. Place a signed custody seal label over the cap such that the cap cannot be removed without breaking the custody seal.

6. Initiate chain-of-custody by designated sampling personnel responsible for sample custody (after sampling or prior to sample packing). Note: If the designated sampling person relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the chain-of-custody prior to this transfer. The appropriate personnel will sign and date the chain-of-custody form to document the sample custody transfer.

II. Packing

1. Using duct tape, secure the outside and inside of the drain plug at the bottom of the cooler that is used for sample transport.

2. Place each sample container or package in individual polyethylene bags (Ziploc® type) and seal.

3. Place one to two inches of vermiculite at the bottom of the cooler as a cushioning material.

4. Place the sealed sample containers and package upright in the cooler.

5. Repackage ice (if required) in small Ziploc® type plastic bags and place loosely in the cooler. Do not pack ice so tightly that it may prevent addition of sufficient cushioning material.

6. Fill the remaining space in the cooler with packing material.

7. Place the completed chain-of-custody forms in a large Ziploc® type bag and tape the forms to the inside of the cooler lid.

8. Close the lid of the cooler and fasten with duct tape.

9. Wrap strapping tape around both ends of the cooler at least twice.

10. Mark the cooler on the outside with the following information: shipping address, return address, "Fragile" labels on the top and on one side, and arrows indicating "This Side Up" on two adjacent sides.

11. Place custody seals over the front right and back left of the cooler lid and cover with clear plastic tape.

III. Shipping

1. All samples will be hand delivered or delivered by an express carrier (e.g., Federal Express) within 48 hours or less from the date of sample collection.

2. The following chain-of-custody procedures will apply to sample shipping:

   a. Relinquish the sample containers to the laboratory via express carrier. The signed and dated chain-of-custody forms should be included in the cooler. The express carrier will not be required to sign the chain-of-custody forms. The sampler should retain the express carrier receipt or bill of lading.

   b. When the samples are received by the laboratory, the laboratory personnel shall complete the chain-of-custody forms by recording receipt of samples, and then check the sample identification numbers on the containers against the chain-of-custody forms.

Appendix C

Field Cleaning/Decontamination Procedures

BLASLAND, BOUCK & LEE, INC.
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Appendix C

Field Cleaning/Decontamination Procedures

I. Materials

- Health and safety equipment (as required in the Health and Safety Plan);
- Distilled water;
- Non-phosphate soap (Alconox® or equivalent);
- Tap water;
- Appropriate cleaning solvent (e.g., nitric acid);
- Rinse collection plastic containers;
- Knife;
- Brushes;
- Aluminum foil;
- Garbage bags;
- Spray bottles;
- Ziploc® type bags; and
- Plastic sheeting.

II. Cleaning Procedures for Small Equipment and Sampling Devices

1. Follow health and safety procedures specified in the Health and Safety Plan.

2. Cleaning of reusable sampling equipment (e.g., scoops, spatulas, etc.), will follow the decontamination procedures presented below:

   a. Non-phosphate detergent and distilled water brush wash;
   b. Distilled water rinse;
   c. Rinse equipment with solvent (dilute nitric acid);
   d. Distilled water rinse; and
   e. Allow to air dry and wrap in aluminum foil.

3. Cleaning/decontamination will be conducted in plastic containers that will be transported to each sampling location. These containers will also be used to collect all decontamination rinsate.

III. Cleaning Procedures for Large Equipment (if applicable)

1. Follow health and safety procedures specified in the Health and Safety Plan.

2. Cleaning of large sampling equipment will follow the decontamination procedures presented below:

   Wash all large equipment with a high pressure water wash using a brush as deemed necessary, to remove any particles.

Attachments

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Attachment A to the QA/SAPP

BBL Quality Assurance Manual

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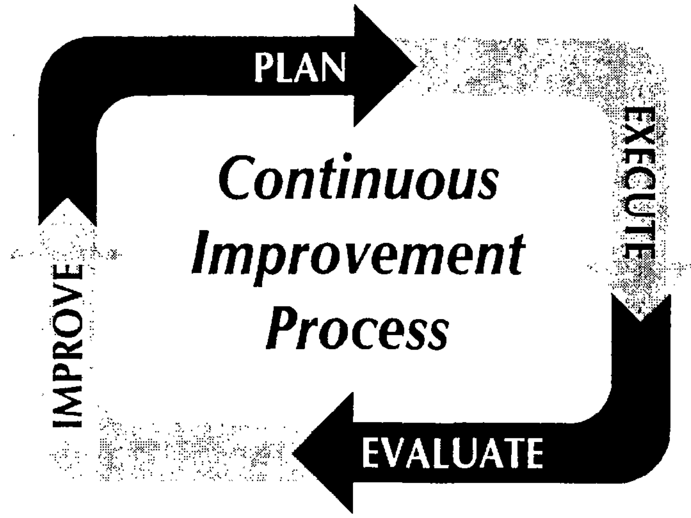
BBL Quality Management Plan

February 2003



Getting from Better to Best

Corporate Quality Officer: ______________ Date: 2/24/03

Legal Counsel: ______________ Date: 2/17/03

President: ______________ Date: 3/3/03

Chief Executive Officer: ______________ Date: 2/27/03

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Quality Policy and Principles

At Blasland, Bouck & Lee, Inc., we have established a Quality Policy and 12 Quality Principles, forming the basis of our continuous improvement activities. The policy and principles are available on the Corporate intranet and are provided below.

QUALITY POLICY	**Continuous Improvement is the basis of our mission to be the "unparalleled provider" of service to our clients. We provide services in a manner that identifies and focuses on each of our client's requirements and expectations. These quality objectives are met by building value-added improvement activities into each and every work effort, not by simply adding quality-related tasks to our normal day-to-day activities. Resources are provided to achieve our quality goals, including but not limited to the development and implementation of training programs and Quality Program assessment activities.**
QUALITY PRINCIPLES	• Quality is the responsibility of all employees, who must strive for continuous improvement, building quality into every activity to produce the unparalleled services our clients expect. • All work activities must be planned based on the client's needs, taking into account quality goals, and applicable technology and regulatory requirements. • Personnel must be qualified to implement the work activities to which they are assigned. Objective evidence of qualifications must be established and maintained. • Procedures must be developed, documented, and approved for project activities. All such work must be performed and documented in accordance with the approved procedures. • Activities involving the acquisition of data must be planned and documented in order to identify the type, quality and quantity of data needed for the intended use. • The procurement and use of materials, equipment, and services that affect the quality of the Firm's (defined on page 4) work must be planned and managed, and must conform to applicable contract, technical, and regulatory requirements. • All designs, plans, specifications, and other documents must be developed using sound engineering and scientific principles, and must meet appropriate industry standards. All designs, plans, specifications, and other documents must be reviewed, verified, and approved prior to issuance. • Complete, accurate, and up-to-date records must be prepared and maintained for all project and program activities. • Sampling, measuring, and testing equipment must be maintained and calibrated in accordance with manufacturer's recommendations and industry standards. Calibration and maintenance records must be maintained. • Computer software and computer hardware/software configurations used in engineering, scientific, and accounting programs must be managed, maintained, and documented. • Deviations from planned project activities must be documented and reported to management as they occur. The significance of a deviation on the project must be determined and appropriate adjustments must be made. • Engineering, scientific, and construction activities will be periodically evaluated to verify conformance with quality, technical, and regulatory requirements.

Table of Contents

1. Quality Management System

Blasland, Bouck & Lee, Inc. (BBL) and its affiliated companies (collectively the "Firm") have established a Quality Program that promotes continuous improvement in the quality of our services. As such, the Firm has established a Quality Management System through which critical-to-quality needs are defined, appropriate resources and personnel are applied, effective procedures are implemented, and other processes for continuous improvement are planned, executed, evaluated, and improved as needed. Through the effective application of our Quality Management System, the Firm strives to provide services that meet or exceed client expectations and regulatory requirements.

The purpose of this Quality Management Plan is to identify the scope of the Firm's Quality Management System and to describe how our processes, people, and resources interact within the system to continuously improve our services.

Collectively, the information and policies identified in this document are referred to as the Quality Management Plan (QMP). The purpose of this plan is to identify the scope of our Quality Management System, to describe the sequence and interaction of the processes included in the Quality Management System, and to identify the policies of our documented procedures. This QMP and associated processes define and communicate the necessary organizational functions and interrelations within the Firm, including responsibilities and authorities for implementing, monitoring, and enforcing our Quality Management System.

This QMP applies to the full range of activities performed by the Firm, whose employees are committed to providing quality services (e.g., reports, letters, work plans, designs and specifications, advice and opinions, data) to our clients. Many of the Firm's activities involve collecting and evaluating data; designing, constructing, and operating systems; and providing management consulting services. The activities that affect quality must consistently meet the intended use, purpose, or scope of work; meet or exceed client expectations; comply with regulatory requirements; be conducted safely; and respect cost considerations.

Our Quality Management system is based upon the core principles of continuous improvement and is guided by various national and international standards, including but not limited to:

- USEPA QA/R2 – Requirement for Quality Management Plans,
- ANSI/ASQ E-4 – National Standard for Environmental Management Systems, and
- ISO 9001:2000 – International Standard for Quality Management Systems.

1.1 Continuous Improvement

At the heart of our Quality Management System is our Continuous Improvement Process (CIP), a mechanism to achieve continuous improvement in all of the Firm's services. This process permeates every aspect of our Quality Management System as we strive to fulfill all of the Quality Principles under the Firm's Quality Policy. Our CIP involves four primary stages, described as:

- ***Planning*** activities are designed to identify the processes needed to provide services and produce deliverables, and to determine the sequence and interaction of these processes;
- ***Execution*** methods provide effective control of these processes and the information necessary to support the work activities;
- ***Evaluation*** methods are in place to measure, monitor, and analyze these processes; and
- ***Improvement*** activities implement actions to achieve planned results or improve processes in an effort to sustain continuous improvement.

1.2 Documentation of the Quality Management System

THE FIRM'S QUALITY MANAGEMENT SYSTEM
Hierarchy of Documentation

- ***Quality Management Plan*** -Occupying the top of the pyramid is this QMP, which declares a core set of policies, procedures, and management responsibilities for developing, executing, and monitoring the Quality Management System as it is applied across the Firm's entire collection of services.

- ***Quality Assurance Procedures*** - Based on the type of services and guided by the Firm's Quality Policy and Quality Principles, the next tier in the system comprises the quality assurance procedures (QPs). The QPs set forth specific policies and requirements for those common processes identified as having the most significant impact on the quality of our Firm's services; they are designed to inform and instruct employees. Selecting appropriate and applicable quality procedures is a management function, resulting in activities that are of the type and quality needed and expected by our clients. QPs are maintained in a central file on the corporate Intranet and are made readily available to all employees.

  The Firm's Quality Assurance Procedures provide "best practice" guidance and are designed to meet EPA QA/R-2 requirements.

  Refer to QP 1.01 for information regarding the control of QPs.

- ***Work Instructions and Standard Operating Procedures*** - The next tier in the system focuses on the project- and program-specific work instructions that promote and guide the delivery of quality services to our clients. These detailed instructions and standard operating procedures (SOPs) are developed and managed both at a corporate level (e.g., *Corporate Policy and Procedures Manual*) and project-, program-, or client-specific level (e.g., SOPs required under a project's Field Sampling Plan or Quality Assurance Project Plan). Through these detailed work instructions, the Quality Management System accommodates the wide array of the Firm's services and the equally wide variety of circumstances under which those services are delivered. In this way, the system remains consistent yet flexible for program-, project-, and client-specific activities (procedures and work instructions) that are applicable to each work effort.

- ***Documentation and Records Control*** - Finally, the Quality Management System is grounded in specific methods and responsibilities for documenting all aspects of the program, including a document and record management procedure. Documented procedures are approved and updated by appropriate personnel, as necessary, prior to issuance. Current revision status of documents are identified, relevant versions of applicable documents are available at points of use, and all documentation remains legible, readily identifiable, and retrievable. Measures are in place to prevent the unintended use of obsolete documents and to apply suitable identification to them if they are retained for any purpose. Documents of external origin are identified, and their distribution is controlled. Records required for the Quality Management System are maintained in order to provide evidence of conformance to requirements, as well as evidence of effective operation of the Quality Management System.

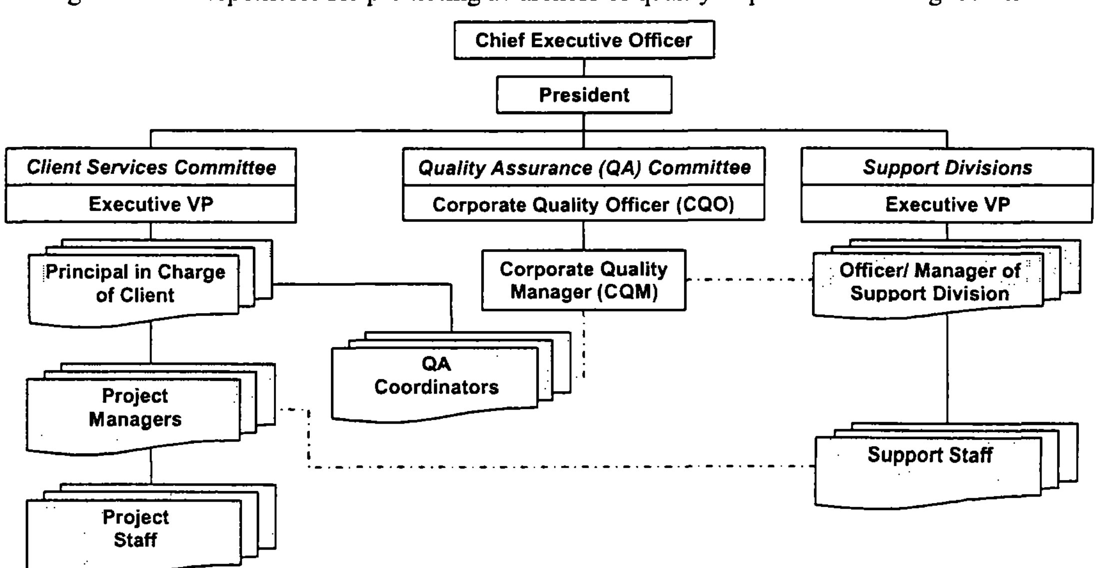
  Refer to QP 1.02 for more information on documentation and records management.

1.3 Management Responsibility and Commitment

Although management provides leadership in implementing the Quality Management System, quality improvement is the responsibility of all employees of the Firm.

The Firm's management is committed to implementation and improvement of the Quality Management System. This is accomplished by setting an example and communicating the importance of meeting client, regulatory, and legal requirements. It is management's responsibility to maintain an atmosphere in which all employees strive to provide quality services and continuously improve. Although managers at the corporate, division, and project levels must lead the implementation of the Quality Management System, quality is the responsibility of all employees. Taken together, the management and all employees of the Firm work as a team to build quality into every project and to deliver the unparalleled services our clients expect.

- ***Organization*** – Located in this section is an abbreviated corporate organizational chart that illustrates the senior management positions responsible for the Firm's Quality Program, including the role of the Corporate Quality Officer (CQO). The Firm's CQO and Corporate Quality Manager (CQM) have the responsibility for overseeing and verifying that processes of the Quality Management System are established and maintained. The CQO and CQM report to senior management on the performance of the Quality Management System and the needs for improvement. The CQO, CQM, and senior management are responsible for promoting awareness of quality requirements throughout the Firm.



As needed and appropriate under this QMP, senior management delegates authority and independence to management personnel and staff, and provide measures to verify that the Quality Management System is understood and implemented by all levels of employees.

- ***Management Review*** - Working closely with the CQO and CQM, the Firm's senior management regularly assesses the adequacy of the Quality Management System, identifies appropriate actions resulting from these assessments, and verifies that corrective actions are completed in a timely manner. Factors that hinder the organization from meeting quality objectives are identified and corrected in a timely manner. For example, at least once each year (or more frequently as deemed necessary by senior management) the Quality Management System and this QMP is reviewed and appropriately updated to reflect changes in the organization, as well as changes in policy and procedures.

  Refer to Section 4.2 of this QMP and QP 4.05 for the procedural aspects of Management Review.

- ***Roles and Responsibilities*** - The following table briefly summarizes the key quality-related roles and responsibilities of management and staff in implementing the Quality Management System.

Summary of Roles and Responsibilities

Personnel	Primary Role in Quality Management System
Senior Management	Accountable to clients, the Firm's Board of Directors, staff, and shareholders. Responsible for promoting, executing, and performing Management Review on all policies identified in this QMP.
Corporate Quality Officer	Accountable to senior management. Responsible for guiding the Firm in the development, execution, and improvement activities associated with this QMP.
Corporate Quality Manager	Accountable to the Corporate Quality Officer. Responsible for managing the Firm's efforts in the execution of this QMP.
Quality Assurance Coordinator	Accountable to the appropriate Principal in Charge (PIC). Responsible for acting as liaison between the CQM/CQO and the client-specific activities that he/she is responsible for.
Principal in Charge of Client (PIC)	Accountable to the client and senior management. Responsible for promoting and executing appropriate and applicable processes identified within this QMP.
Project Manager	Accountable to the PIC. Responsible for understanding all processes identified in this QMP, for identifying all applicable processes for use on his/her project, and for communicating and verifying that all project team personnel are following documented processes.
Staff	Accountable to the Project Manager and individual supervisor. Responsible for performing activities in accordance with stated processes identified within this QMP.

The Firm's senior management is committed to providing the resources required for the effective planning, execution, evaluation, and improvement of services and work procedures under our Quality Management System. Such critical-to-quality resources include, but are not limited to, human resources, training, and facilities:

- ***Human Resources*** - Personnel who are assigned responsibilities defined in the Quality Management System are competent on the basis of applicable education, training, skills, professional licensing, and experience. A performance evaluation is performed for all staff on an annual basis. Supervisors are responsible to continually identify additional skills needed by personnel performing activities affecting quality. Education and/or training is provided to fulfill these needs, and the effectiveness of this training is evaluated by the Supervisor. Supporting feedback is provided from appropriate project management personnel. Supervisors are also responsible for identifying retraining needed for their personnel. Employees are aware of the relevance and importance of their activities and how they contribute to the achievement of the quality objectives. Appropriate education, experience, training, licensing, and qualification records are maintained.

- ***Education/Training*** – Our corporate learning environment encourages all employees to actively participate in career-building education and training programs. This is achieved by providing a balanced, continuously improving, and interactive education and training program for all employees to enhance personal growth, technical knowledge, and client focus.

  Training needs are identified through individual competency evaluations. These needs are met through corporate-directed programs. The Firm requires specific levels of training and education for each employee depending on their position and role. Quality-specific training for all of the Firm's employees is provided annually. To engage in training activities and events, employees obtain approval from their Vice President. After completion of the training, employees are required to complete an evaluation to assess the relevance and effectiveness of the training. More formal education activities are supported through the Firm's tuition reimbursement program, which applies to both degree/certificate education and individual college-level courses. Education and training records are maintained for each employee.

- ***Facilities*** - Human and physical factors of the work environment, including workspace, associated facilities, equipment, hardware, software, and supporting services, are identified, provided, and maintained for achieving quality services and continuous improvement of those services.

2. Planning Activities

Planning activities begin with the identification of client requirements and the establishment of quality objectives for these requirements. Additional activities include the identification of applicable procedures and the need to establish processes and documentation and the provision of resources and facilities specific to client requirements.

Our ability to meet and exceed client expectations is dependent upon successfully planning and executing projects. Project planning develops the concept and approach, identifies the client's quality requirements, and makes provisions for continuous improvement through monitoring and evaluation throughout all phases of the project's life.

Applicable Quality Procedures (QPs) identify the processes needed to meet or exceed client requirements and to review, verify, and validate activities appropriate to each design and/or development stage, as well as the responsibilities and authorities for these activities. Interaction between different groups involved in planning, design, and development is actively managed for effective communication and understanding of responsibilities. As activities progress, planning activities are updated as part of the CIP.

The Firm's *Project Management Handbook* provides numerous essential tools for successful planning and implementation of projects. Coupled with the Client Quality Plan (CQP) developed for each client, the *Project Management Handbook* is intended to facilitate consistent application of good planning, execution, evaluation, and improvement practices according to this QMP and associated specific QPs.

2.1 Project Planning

It is our policy to successfully plan and execute each and every project, enabling us to meet or exceed our clients' expectations. Projects are planned to facilitate efficient project implementation (from the development of concept and approach through project completion) and to identify the client quality requirements for the proposed work. Projects are monitored for changing conditions throughout all phases of the project's life and are periodically reviewed to determine whether appropriate steps have been taken to meet project goals and to evaluate the effect of any changes or non-conformances.

Refer to the Project Manager's Handbook *and QP 2.01 for information on project planning.*

2.2 Procurement of Products and Services

It is our policy to plan for and then procure all the goods and services required to conduct our business activities in accordance with fair, ethical, and legal trade practices. Based on sound planning and project management, goods and services are procured when and where needed, in and of the desired quantity and quality, at the lowest possible price, via the most economical shipping methods, and by personnel authorized to execute procurements on behalf of the Firm.

Refer to QP 2.02 for information regarding procurement requirements.

2.3 Control of Changes

When requirements are changed, relevant documentation is amended, and affected personnel are made aware of said changes. Changes are identified, documented, and controlled, including evaluation of the effect of the changes on the services provided. The changes are verified and validated, as appropriate, and approved before implementation. Results of the review of changes and subsequent follow-up actions are documented.

Refer to the Project Manager's Handbook *and QP 2.01 for guidance on controlling project-related changes.*

2.4 Control of Measuring, Testing, and Monitoring Devices

The Firm uses a wide range of measuring and test equipment in the course of its activities. To maximize the quality (i.e., accuracy, precision, usefulness) of data collected from these devices, equipment used by the Firm (and subcontractors) must be in the condition required for the performance of specified activities. Documented procedures for performing and documenting calibration and for the preventive maintenance of measuring and test equipment are followed to provide necessary controls. Under the Firm's Quality Management System and according to applicable project-specific Field Sampling Plans (FSPs) and/or Quality Assurance Project Plans (QAPPs), measurements and the measuring, testing, and monitoring devices required for verifying conformity to specified requirements must be identified. Measuring and monitoring devices are then used and controlled to verify that measurement capability is consistent with the measurement requirements.

Refer to QP 2.04 for information on measuring, testing, and monitoring devices.

2.5 Development of Client Quality Plans

It is our policy to treat each client as the most important client we have. Therefore, effective client-focused planning is essential for meeting or exceeding the quality expectations of our clients. Each client-specific Principal in Charge (PIC) identifies and establishes quality measurements and monitors the most significant client expectations. Each PIC oversees development of a CQP that provides a tool for developing and implementing a program to meet or exceed client expectations. Each CQP should be considered a "living" document and, therefore, be designed and periodically reviewed to allow adaptation to changing client requirements and expectations.

Refer to QP 2.05 for information on the content and role of the CQP.

2.6 Proposal Development

It is our policy that all proposals and qualification documents accurately represent the Firm's ability to provide services for the client and meet applicable regulatory requirements.

Refer to QP 2.06 for information on proposal development.

2.7 Management of Computer Hardware and Software

The Firm employs various computer hardware components and software programs in managing and implementing client projects. The Firm's Computer Information Technology (CIT) group is responsible for all computer-related activities, including but not limited to servers, desktops, networks, applications, and web technology. It is the responsibility of the CIT group to test, install, maintain, control, and document computer hardware and software used by staff to complete projects. CIT assists staff in assessing user requirements, testing new technologies and programs, and recommending and purchasing hardware and software to meet each user's needs. Of primary importance to the Firm is the security and privacy of all information contained in digital (electronic) form.

All new hardware or software used for project activities follows a process that verifies that the needs of the end user are being met. This includes commercial off-the-shelf solutions, as well as custom solutions built in-house. This begins with a defined statement of work (SOW) that identifies the task to be performed, assumptions, dependencies on other systems, the end user, and the test/delivery criteria. The SOW must be approved by the Project Manager prior to implementation. As appropriate, a detailed specification is created to provide additional details of the system to be deployed. This specification is reviewed and approved by the appropriate Project Manager.

Upon deployment, logs are maintained to reflect significant events and changes to hardware configuration. Software changes are managed through a change process appropriate to the scope of the change. Simple "bug" fixes are made at the discretion of the designer/developer, while major changes follow the statement of work/specification process described above.

Note: For activities specific to data management, refer to QP 3.09.

3. Execution Activities

Execution activities include the sequence of processes and sub-processes involved in providing services that meet or exceed client expectations. These activities are controlled through information that specifies the characteristics of the service and the availability of work instructions, where necessary. The services provided by the Firm are measured and reviewed at appropriate stages to verify that requirements are met. Evidence of conformity with the acceptance criteria is documented, and records indicate the authority responsible for release of services and/or deliverables. Services and/or deliverables are not released until all the specified activities have been satisfactorily completed, unless otherwise approved by the client.

Quality-related processes necessary for providing services to our clients are planned and managed. These "living" processes not only provide consistency, but also allow *for continuous improvement* within the framework of the Quality Management System.

- ***Review/Verification of Services*** - Technical reviews of services, designs, and/or documents are conducted to evaluate the ability to fulfill requirements and to identify problems and propose follow-up actions. Reviews are conducted by appropriate representatives independent of the discipline associated with the activity being reviewed. Results of reviews and subsequent follow-up actions are recorded. Action is taken to verify that the service provided meets the intended goals.

- ***Validation of Services or Processes*** - Validation confirms that the result of the service meets its intended use. When applicable, validation is completed prior to the delivery or implementation of the service. When this is impractical, partial validation is performed to the extent applicable. Results and subsequent follow-up actions are recorded. Applicable validation includes the qualification of processes, equipment and personnel, the use of defined methodologies and procedures, the requirements for records, and re-validation.

- ***Client Property*** - Care of client property is exercised while such property is in the Firm's control or use by identifying, verifying, protecting, and maintaining the client property provided for use or incorporation into a deliverable. This may include but is not limited to intellectual property or confidential information. Incidents in which client property is lost, damaged, or otherwise found to be unsuitable for use are recorded and reported to the client.

3.1 Technical Document Process

It is our policy to produce technical documents that are of consistent high quality. Documents must fulfill client and regulatory requirements and be technically accurate and legally defendable. Six main steps encompass the technical documents process: planning, executing, evaluating, approving, delivering, and managing technical documents and records.

Refer to QP 3.01 for guidance on the technical document process and to the Document Standards Guide *for information on proper document formatting and numbering.*

3.2 Development and Verification of Figures, Tables, and Logs

It is our policy to produce graphic representations of information that are of high quality. Figures, tables, and logs must fulfill client and regulatory requirements and be technically accurate and legally defendable.

Refer to QP 3.02 for information on developing and verifying figures, tables, and logs.

3.3 Preparation of Calculations

Our policy dictates that calculations be documented to a degree that a technical peer who may not be familiar with the project could understand the methodology, assumptions, justification, and references used. The goal of the calculation process is to have a minimum of two technically qualified individuals agree that the information presented in the calculation is accurate and is documented in sufficient detail.

Refer to QP 3.03 for guidance on preparing and reviewing calculations.

3.4 (Section intentionally left blank)

3.5 Documentation of Field Activities

It is our policy that all field activities be documented to show compliance with projects plans, work plans, and contract terms and to serve as evidentiary records. Documentation of activities must be legible, organized, and complete. All fieldwork documentation must include, at a minimum, project title and number, date and time of activities, identification of the employee performing the work, and the specifics of the work.

Refer to QP 3.05 for information on documenting field activities.

3.6 Field Sampling Activities

It is our policy that field sampling, measurements, and observations be conducted in accordance with approved site-specific planning documents. These activities must be documented to provide an evidentiary record and to demonstrate that such activities have been performed properly. Applicable documents include work plans, the Quality Assurance Project Plan (QAPP), the Field Sampling Plan (FSP), applicable standard operating procedures (SOPs), the Health and Safety Plan (HASP), and other appropriate project documents associated with the sampling program.

Refer to QP 3.06 for information on requirements pertaining to sampling activities.

3.7 Construction Inspection and Observation Activities

It is our policy to perform construction inspection and observation services in a manner that is focused on client needs while maintaining appropriate risk management.

Refer to QP 3.07 for information on construction inspection and observation activities.

3.8 Development of Design Documents

It is our policy that all engineering design documents (design reports letters, contract drawings, technical specifications, performance specifications, and pre-purchase specifications) produced by the Firm be developed under the guidance of, and be signed by, the designated Engineer of Record (EOR) or authorized professional expert for the appropriate discipline. The Firm's design projects shall be technically sound (as defined by industry standards of care) and meet the client's goals and objectives, as well as applicable local, regional, state, and federal requirements.

Refer to QP 3.08 for information on developing design documents.

3.9 Data Management

It is our policy that data management activities follow a common process to establish and meet data quality objectives in compliance with client requirements and federal and state regulations. A typical BBL project entails acquisition, interpretation, and management of data.

Refer to QP 3.09 for information on data management.

3.10 Design During Construction Activities

It is our policy that all engineering efforts during design and construction activities be technically sound, meet the client's requirements, meet all applicable (local, regional, state, and federal) regulations, and maintain appropriate risk management.

Refer to QP 3.10 for information on design during construction activities.

3.11 Operation, Maintenance, and Monitoring Activities

It is our policy to provide our clients with operation, maintenance, and monitoring (OMM) services that comply with applicable regulations, are conducted safely, and performed efficiently on a consistent basis. Our goal is to provide value-oriented solutions that provide cost savings, reduced liabilities, and other positive business outcomes for our clients.

Refer to QP 3.11 for information on operation, maintenance, and monitoring activities.

4. Evaluation and Improvement Activities

Continuous Improvement is facilitated through the use of this Quality Management Plan, the Firm's Quality Policy and Principles, assessment results, corrective and preventive action, employee input, and management review.

Processes necessary for the continuous improvement of the Quality Management System are planned and managed for the identification and execution of improvement opportunities. Nonconformities and/or services that do not conform to intended requirements are identified, controlled to prevent unintended use or delivery, and corrected. When nonconforming services are detected after delivery or use has started, appropriate action is taken based on the consequences of the nonconformity. Proposed rectification of the nonconformance is reported to the client, end-user, regulatory body, and others, as appropriate.

4.1 Corrective and Preventive Action

It is our policy to take corrective action to eliminate a defect from our work activities, and to confirm that the action taken is appropriate to the impact of the problems encountered. Preventive action is taken to eliminate the causes of potential nonconformities in order to prevent occurrence. A QP exists for identifying nonconformities, determining the causes of nonconformity, evaluating the need for actions to verify that nonconformities do not recur, determining and implementing the corrective and/or preventive action needed, recording results of action taken, and reviewing the corrective and/or preventive action taken.

Refer to QP 4.01 for information on corrective and preventive action.

4.2 Quality Assurance Assessment

Assessments of programs, projects, and specific project activities are conducted, and the results are evaluated to measure the effectiveness of the Quality Management System. Such assessments may involve both management and technical reviews by individuals involved in the program, and also by those independent of the work.

- ***External Assessment*** - A client satisfaction survey process is performed on an annual basis to solicit client perceptions of the services that the Firm provides.

- ***Internal Assessment*** - Internal assessment is used to verify that the service activities performed and provided to our clients conform to the intended purpose and requirements defined in the QMP and the related QPs. Three approaches are used to perform internal assessments: internal audits, mini-reviews, and self-assessments.

- ***Management Review*** – Senior management reviews the inputs and outputs of the Quality Management System at planned intervals to verify its continuing suitability, adequacy, and effectiveness. The review evaluates the need for changes to the organization's Quality Management System, including the quality policy, quality objectives, and procedures. These reviews are recorded.

Refer to QP 4.05 for information on assessment activities.

Attachment B to the QA/SAPP

Laboratory Quality Assurance Manuals

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Marine Sciences Laboratory

EFFECTIVE DATE: 4-25-00

Battelle Pacific Northwest Laboratories
Marine Sciences Laboratory

STANDARD OPERATING PROCEDURE

MSL-A-001-04

SAMPLE LOG-IN PROCEDURE

Approvals:		
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	Signature	Date
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	Signature	Date

SAMPLE LOG-IN PROCEDURE

1.0 SCOPE AND APPLICATION

This method applies to sample receipt, log-in, preservation, storage, and disposal of all chemistry water, soil, sediment and tissue samples by the Battelle Marine Sciences Laboratory (MSL) Sample Custodian or designee.

2.0 DEFINITIONS

Log-In - The procedure by which samples are received and documented at the (MSL).

Sample Custodian - The person responsible for sample receipt and log-in.

CoC Form - Chain-of-custody form, accompanies samples from field to MSL and is kept with the sample files.

3.0 RESPONSIBLE STAFF

Sample Custodian or designee
Project Manager
Data Coordinator
Quality Assurance Officer or Representative

4.0 PROCEDURE

4.1 Sample Receipt

4.1.1 Samples arrive from the customer via a variety of delivery systems (e.g., United Parcel Service, Federal Express, Air Borne, Courier Service, General Delivery or from staff within MSL). The Sample Custodian is notified of sample arrival by either the shipping clerk or directly by the MSL staff member who has custody of the samples. The Log-In Checklist (see Attachment 1) is initiated.

4.2 Sample Log-In

4.2.1 The shipping container is inspected for a custody seal and opened. The container temperature is taken immediately and recorded on the Log-In Checklist. A calibrated thermometer or temperature probe (see MSL-W-003, Calibration and Use of Thermometers, for calibration instructions) is placed in the cooler in a representative location (not directly touching any ice or cold packs). If a temperature blank sample is provided, this should be used to measure the container temperature at the time of receipt. Acceptable shipping temperatures are less than or equal to room temperature for metals in preserved water samples. For unpreserved water, tissue, sediment, or soil samples, container temperature(s) should be 4°C $\pm$ 2°C; however, solid samples (tissue, sediment, and soil) can be frozen.

4.2.2 Individual samples are then removed from the shipping container and inspected for the presence of sample custody seals, the intactness of the seals, damage, leakage, missing labels and/or other problems. These problems are noted on the Log-In Checklist. The sample identification numbers are compared to the CoC form that accompanies the samples to ascertain whether all samples are present and whether or not the labels on the containers match those on the CoC form. If no CoC form accompanies the samples, a MSL CoC form will be initiated and filled in with the sponsor codes listed on the sample labels.

4.2.3 At this point, the Project Manager should be notified of the number and condition of the samples (i.e., if any seals have been broken, sample containers are damaged, and/or the cooler temperature(s) are out of limits). The project manager is responsible for contacting the customer regarding problems that may affect the integrity of the samples or subsequent analyses. The discrepancy, its resolution, and the identity of the person contacted must be documented in the project file.

4.2.4 After complete inspection of the samples, the "Received By" section of the CoC form must be signed and dated, along with the receipt time, by the Sample Custodian.

4.2.5 From the Project Log-In/Central File Index (Log-In book), a project or initial set of samples is assigned a sequential central file number (CF#). The log-in book is a spiral notebook in MSL-5, Room 130. Subsequent sample sets received for ongoing projects are logged in with the same central file number. A group of samples can be one of several things: a set of samples from one sampling period or a continuous influx of samples from one project. The project manager usually determines this.

4.2.6 Each sample is then assigned an individual number after the central file number (e.g., 1205*1, 1205*2, etc...). Samples from an ongoing project are numbered beginning with the last number assigned. This number is called the MSL sample ID, and is recorded on the CoC form and on the corresponding sample container. All samples are referred to by the MSL sample ID from this point on until the data are reported, when the sponsor ID is matched back to the MSL sample ID.

4.2.7 The Sample Custodian records in the log-in book (see Attachment 2) the central file number, date samples are received, file name, project sample description, samples numbers, storage location and the custodian's initials.

4.3 Sample Filtration, Preservation and Storage

4.3.1 Samples are appropriately preserved to ensure sample holding times, depending on the matrix and analyses. In addition, container type and amount of sample are observed to make sure the integrity of the sample is maintained. Tables 1 and 2 provide recommended practices for sample preservation and container types. If there are any deviations, the deviation shall be documented and the Project Manager should be notified for direction.

Table 1. Preservation Recommendations

Analysis Requested	EPA 1600 Series and EPA 245 Suggested Practices	MSL Recommended Practices	Holding Time
Metals in water - except methyl Hg, Cr^{3+}, and Cr^{6+}	Acidify with ultrapure nitric acid to pH<2 (0.2%)	2mL concentrated ultrapure nitric acid/1L sample (0.2%) to pH<2	6 months
Total Hg in water	Acidify to 0.5% with ultrapure HCL or BrCl to pH<2 (0.5%)	5mL concentrated ultrapure HCl/1L sample (0.5%)	28 days
Methyl Hg in water	4 mL concentrated ultrapure HCl/1L freshwater sample or2mL concentrated sulfuric acid 1L seawater sample	5 mL concentrated ultrapure HCl/1L freshwater sample or 5mL 8M sulfuric acid/1L seawater sample	28 days
Cr^{3+} water samples	Add 1mL Cr^{3+} extraction solution to 100mL sample, vacuum filter through 0.4μm membrane, add 1mL concentrated ultrapure nitric acid to filter	Add 1mL Cr^{3+} extraction solution to 100mL sample, vacuum filter through 0.4μm membrane, add 1mL concentrated ultrapure nitric acid to filter	6 months
Cr^{6+} water samples	1mL 50% NaOH 125mL sample and refrigerate	1mL 50% NaOH /125mL sample and refrigerate	30 days
Arsenic speciation in water	Acidify pH<2 with HCl and refrigerate	2mL concentrated ultrapure HCL/1L sample (0.2%) and refrigerate	28 days

Table 1. Preservation Recommendations (continued)

Analysis Requested	EPA 1600 Series and EPA 245 Suggested Practices	MSL Recommended Practices	Holding Time
Selenium speciation in water	Not applicable	2mL concentrated ultrapure HCL/1L sample (0.2%) and refrigerate	28 days
Metals in tissue, sediment, and soil	Ship cold (4°C ±2°C) then freeze dry	Freeze and/or refrigerate (4°C ± 2°C) then freeze dry	6 months
Total Hg in tissue, sediment, and soil	Ship cold (4°C ±2°C) then freeze dry	Freeze and/or refrigerate (4°C ± 2°C) then freeze dry	28 days
Methyl Hg in tissue	Ship cold (4°C ±2°C) then freeze dry	Freeze and/or refrigerate (4°C ± 2°C) then freeze dry	28 days
Methyl Hg in sediment	Ship cold (4°C ±2°C) DO NOT FREEZE DRY	Freeze and/or refrigerate (4°C ± 2°C) DO NOT FREEZE DRY	28 days

Table 2. Container Types and Minimum Samples Amount

Analysis Requested	Container Type	Minimum Required Amount
Metals in water (except mercury)	fluoropolymer (FEP; Teflon™), polyethylene, polycarbonate, or polypropylene bottles with lids	150 mL
Metals in sediment and tissue	glass, polyethylene, polystyrene (SPEX) jars	10-20 g wet
Total and methyl Hg in water	fluoropolymer (FEP; Teflon™) or glass bottles with fluoropolymer or fluoropolymer-lined lids	500 mL
Total and methyl Hg in sediment and tissue	fluoropolymer (FEP; Teflon™), glass, or polystyrene (SPEX) jars	10-20 g wet

4.3.2 If water samples arrive unpreserved, the project manager is consulted to determine if filtration or preservation is required. Sample pH may be randomly measured to assure that samples are not preserved when other information is unavailable. Water samples have to be filtered before preservation with acid. Once filtration is complete, samples are acidified and all is noted on the Log-In Checklist form.

If the samples are to be analyzed for the Navy and are water samples for metals or mercury (Hg) analysis, the pH (see MSL-W-001, Calibration and Use of pH Meters) for all samples will be measured and the result and time of measurement documented using the form found in MSL-I-028, Navy Sample Analyses Plan. The pH of all other water samples for metals will be determined based on customer request.

4.3.3 Tissue, sediment and soil samples can be held in a refrigerator or freezer until sample preparation. If samples require freeze drying as per Project Manager instructions, samples are weighed and placed in the ultra-low temperature freezer ($-68 \pm 5\,^{\circ}C$) located in MSL 5, Room 130.

4.3.4 All samples are placed in the location specified in the sample log-in book by the sample custodian. At this time, the Log-In Checklist is completed, signed and dated by the Sample Custodian.

4.3.5 Additional project information must be obtained from the project manager prior to the electronic log-in of the samples. This includes the customer name and sample analyses required. At this point, the sample information will be used to generate a computer spreadsheet of the sample log-in information called the Sample Log-In spreadsheet (see Attachment 3). A copy of the Sample Log-In form along with the completed Chain-of-Custody form(s), Log-In Checklist, and any custody information that accompanied the samples should be given to the Project Manager accompanied by a kit/addendum (see MSL-D-004, Data Reporting, Reduction, Back Up, and Archiving). The Project Manager will complete the kit/addendum and forward the packet to the Data Coordinator. The Data Coordinator will disperse copies of the kit/addendum to the appropriate analysts.

4.4 Sample Disposal

Sample disposition can either consist of returning samples to the customer or disposing of samples into an appropriate waste receptacle. Sample disposition requirements are documented on the Log-In Checklist. MSL sample disposition is mandated by the formal, Battelle Pacific Northwest National Laboratory-controlled document issued under the Standards Based Management System (SBMS) in the subject area of "Chemical Management System". The SBMS is a web-based policy and procedures resource for Battelle staff, which guides day-to-day operations. The Chemical Management System (CMS) tracks solution disposal (e.g., for samples, reagents, standards, etc.) Samples are labeled as to their disposition date when archived. A Sample Disposal Logbook is used to document Sample Disposal.

5.0 DATA ANALYSIS AND CALCULATION

Not applicable to this procedure

6.0 QUALITY CONTROL

The Project Manager reviews the CoC and Log-In Checklist for correctness and completeness before the kit/addendum is completed. When the QA reviews the data, the CoC and Log-In Checklist are reviewed again for correctness and completeness.

7.0 SAFETY

Precautions should be used when handling samples. Gloves, safety glasses and laboratory coats should be worn when handling samples of unknown content.

8.0 TRAINING REQUIREMENTS

Appropriate health & safety training is required to handle hazardous samples. All staff members who will be designated as a Sample Custodian shall first read this procedure. Documentation of training will be recorded on a training assignment form from MSL-A-006, Marine Sciences Laboratory Training.

9.0 REFERENCES

MSL-W-003	Calibration and Use of Thermometers
MSL-W-001	Calibration and Use of pH Meters
MSL-I-028	Navy Sample Analyses Plan
MSL-D-004	Data Reporting, Reduction, Back Up, and Archiving
MSL-A-006	Marine Sciences Laboratory Training

Attachment 1
Example Log-In Checklist

LOG-IN CHECKLIST Reference SOP# MSL-A-001

Central File #: ____________ Project Manager: ____________

TO BE COMPLETED BY PROJECT MANAGER (prior to sample arrival when possible)

Matrix: ____________ WP# ____________

Yes	No	
☐	☐	Navy-type Project (requires high-level sample tracking procedures)
☐	☐	Filter Samples: ***Amount:*** Entire sample Half of sample
☐	☐	Freeze dry sample(s) - samples will be weighed and placed in Ultra-low temp freezer (Lab# 130)
☐	☐	Special instructions: ____________

Sample Preservation Instructions: ____________

Date To Archive: ____________ Date To Dispose: ____________

TO BE COMPLETED UPON SAMPLE ARRIVAL/LOG-IN

Yes	No	N/A	Initial Appropriate Box When Completed
☐	☐	☐	Custody seal present
☐	☐	☐	Custody seal intact
☐	☐	☐	Cooler(s) temperature (acceptable range 4°C±2°) ____________ °C (if multiple coolers, note temp. of each) ____________ °C
☐	☐	☐	Project Manager notified of any discrepancies or if temperature outside acceptable range? Comments/Remedy: ____________
☐	☐	☐	All chain of custody forms signed and dated?
☐	☐	☐	Samples Filtered?

Sample condition(s): **Acceptable** **Other (explain):**

Container type: **Teflon** **Poly** **Glass** **Spex** **Other:** ____________

Notes: ____________

Completed By: ____________ **Date/Time:** ____________

Attachment 1
Example Log-In Checklist (continued)

SAMPLE PRESERVATION

☐ Sample preserved upon arrival at MSL (noted on CoC / Sample / per PM Instruction)

☐ Random pH checked for ~10% of samples (use dip paper) Sample IDs: ________________

☐ Complete pH check required for project (use pH meter and record on pH Record form)

If preservation necessary, record Acid Lot#

Type: ☐ 0.2% HNO3 Notes: ________________________________

☐ 0.5% HCl (Hg samples) Notes: ________________________________

☐ Refrigerate Notes: ________________________________

☐ Freeze Notes: ________________________________

☐ Other Notes: ________________________________

Completed By: ________________________ **Date/Time:** ________________

Attachment 2
Example Project Log-In/Central File Index (Log-In Book)

DATE	INITIALS	FILE NAME	PROJECT	SAMPLE DESCRIPTION	# OF SAMPLES	STORAGE LOCATION

Attachment 3
Example Sample Log-In Spreadsheet

cc: PM/CF Log-In File	**Project Title:**	**Example Sample Log-In Sheet**			**Project Manager:** **Date Received:**	
SPONSORR CODE	Battelle MSL Sample ID	Matrix	Storage Location	Parameters requested	**Collection Date**	Initials
Dump Site 1	***1205*1***	***Sediment***	***Hg Lab***	*MeHg*	***02/29/99***	***LN***

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Marine Sciences Laboratory

EFFECTIVE DATE: 4-17-03

Battelle Pacific Northwest National Laboratories
Marine Sciences Laboratory

STANDARD OPERATING PROCEDURE
MSL-I-016-06
TOTAL MERCURY IN TISSUES AND SEDIMENTS BY COLD VAPOR ATOMIC ABSORPTION (CVAA)

Approvals:		
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	Signature	Date
QA OFFICER: Deborah Coffey	Deborah Coffey	4/17/03
	Signature	Date
TECHNICAL GROUP MANAGER: Eric Crecelius	Eric Crecelius	4/17/03
	Signature	Date

TOTAL MERCURY IN TISSUES AND SEDIMENTS BY COLD VAPOR ATOMIC ABSORPTION (CVAA)

1.0 SCOPE AND APPLICATION

This method is applicable to the determination, in parts per million, of total mercury (Hg) in acid-digested sediment and tissue samples by cold vapor atomic absorption (CVAA). This procedure replaces Battelle procedure, MSL-M-031, and it is a modification of EPA Methods 245.5 and SW-846 7471A. The modification is in the digestion, because the EPA digestions uses potassium permanganate, which is a source of Hg contamination. This method uses the digestion method outlined in the NOAA Technical Memorandum NOS ORCA 130 "Sampling and Analytical Methods of the National Status and Trends Program Mussel Watch Project: 1993-1996 Update". G.G Lauenstein and A.Y. Cantillo, eds.

2.0 SUMMARY OF METHOD

Mercury ions in a digestate are reduced by acidic $SnCl_2$ to Hg^+, then carried through a flow cell on a stream of inert gas, i.e. argon. A photometric detector measures the luminous intensity of monochromatic light that has passed through the sample and compares it with the luminous intensity of the same light that has passed through a reference beam. Mercury atoms absorb light at 253.7 nm. The attenuation of the light is directly proportional to the concentration of the mercury vapor, which is quantified using a standard curve. The typical detection limit for the method is 0.001 ug/g.

3.0 RESPONSIBLE STAFF

- Technician - sample digestion
- Analyst- sample analysis
- QA Officer or Representative- data verification

4.0 PROCEDURE

4.1 Sample preparation

Samples shall be digested using an appropriate strong acid digestion, which results in complete sample dissolution. It has been found that the traditional potassium permanganate digestion used in many standard methods for mercury analysis results in unacceptably high and inconsistent blank levels and should be avoided. Digestions done in a closed Teflon® vessel (bomb) are recommended, as many open vessel and microwave techniques result in loss of Hg during venting.

4.2 Apparatus and Reagents

4.2.1 Thermo Separation Products "mercuryModule" (mercury vapor generator).

4.2.2 Thermo Separation Products "mercuryMonitor 3200" (elemental mercury detector).

4.2.3 Thermo Separation Products "autoMetric 3000" (autosampler).

4.2.4 Computer with HgTalk program, screen and printer.

4.2.5 Rinse system for sample siphon - tubing, rinse water bottle, waste water bottle and flow regulating valve.

4.2.6 Acid cleaned (cold 50% HNO_3 for at least 2 days) test tubes, 13 X 100 mm.

4.2.7 Hydrocarbon trap for purifying gas flow into instrument.

4.2.8 Drying trap (4-8 mesh, reagent soda lime is preferred over the magnesium perchlorate that was recommended by the company. The soda lime lasts longer, is less hazardous, and causes less interference).

4.2.9 Pre-pure Argon gas.

4.2.10 Stannous chloride, 10% - add 50 g of $SnCl_2$ to 100 mL low Hg water and 50 mL of HCl in a specially marked Teflon bottle then fill to 500 mL with low Hg water. Bubble nitrogen gas through mixture for at least 6 hours at a very low flow. Transfer solution to 2 specially marked brown bottles. 2% $SnCl_2$ - dilute 50 mL of this 10% $SnCl_2$ with 200 mL of 10% HCl.

4.2.11 Nitric Acid, 3% - add 60 mL of HNO_3 to low Hg water and bring to 2L volume.

4.2.12 Mercury stock standard, 1000 mg/L - purchased from High Purity Standards, Inc.; expiration is usually 1 year.

4.2.13 Mercury intermediate standard, 10 mg/L - dilute 1 mL of 1000 mg/L stock standard into 100 mL of 1% HCl and store at room temperature in a Teflon bottle dedicated for use with the intermediate standard. As long as the standard is stored in Teflon, the standard will be stable for a period of at least 3 months and expiration can be expected to be the same as the stock standard.

4.2.14 Mercury working standards, dilute the intermediate standard (10 mg/L Hg) into 5 concentrations (i.e. 0.5 ug/L, 1.25 ug/L, 5.0 ug/L, 7.5 ug/L and 12.5 ug/L) and store at room temperature in Teflon bottles dedicated for use with standards. As long as the standards are stored in Teflon, the standards will be stable for a period of at least 6 months and expiration can generally be expected to be the same as the stock standard. Check the standards against the certified reference materials (CRMs) run in each analytical batch to verify that the working standards are retaining their titre.

4.3 Interferences

4.3.1 Take appropriate precautions to prevent stannous chloride contamination during sample handling or of any equipment associated with this analysis. If contamination should occur, mercury will be volatilized and lost.

4.3.2 Particle size in samples must be less than 10 μm.

4.3.3 Samples must be totally digested or organic material will interfere with detection.

4.4 Analysis

This analysis is entirely computer based. Software use and troubleshooting will not be reiterated in this procedure, because this information can be found in the software manual.

4.4.1 Allow the mercury monitor at least one hour to warm up before analysis. Turn on the mercury generator, the printer and especially the autosampler before the HgTalk program is loaded or the connection will not be made.

4.4.2 Change the soda lime trap after approximately 100 samples or if the analyzer has not been run for several days. Check the reagent bottles for proper amounts, empty the hazardous waste bottle and the rinse waste bottle, then turn on the argon gas to a pressure of 80 psi. Make sure the reagent pressure gauge reads 5.0 ±1 psi (regulator is on the rear panel) and the flow meter reads about 0.2.

4.4.3 Open HgTalk. There are 3 files necessary for analysis; "method", "sequence", and "data". The method file contains the calibration, integration parameters, and the reports possible. The method file currently being used is called "Hgmeth.hmh". The sequence file is the operation and calculation of the analysis and must be created and named for each analysis set. Each sample has its own data file, which is created during the analysis.

4.4.4 After entering HgTalk and checking that the information in the method file is correct, a sequence file must be created. It is best to have the information organized and calculated before entering the information into the file since this program does not multiply dilution factors or allow the creation of new columns. With the 5 ml loop in the mercury generator, there should be at least 6 ml of sample in the test tube, which does not hold more than 9 ml. The sample can be straight digestate, however this usually does not leave enough digestate for another analysis. 3 mL of digestate and 3 ml of 3% HNO_3 or 1 mL of digestate and 5 mL of 3% HNO_3 are the common dilutions used currently. With the information of sample, weight, and volume times dilution factor ready (see attachment #1 for an example), the new sequence can now be created.

A. Open "edit sequence" and choose "new".
B. Fill in "sequence header" with method file name, create new data file name, operator name and date.
C. Choose "edit", "create entries", answer "yes" to function overwrite, and fill in number of standards and samples.
D. Fill in the sequence spreadsheet that appears with requested information. See Attachment #2 for an example.
E. Choose "SaveAS" and name file. Exit.
F. In the first 6 sample tubes, place a calibration blank and each of the five working standards. These samples will be used to generate the calibration.

G. Pipette the correct volume of 3% HNO_3 into clean test tubes and add appropriate amount of digestate to achieve the desired dilution. Rinse pipette tip several times in the resulting solution. Be sure to pour the correct standard into the vials numbered in the sequence file. Check that the sample vials match the vial numbers in the sequence. Place the tray on the autosampler and check the flow of the rinse water.

H. Load file and choose "run", then "start".

4.4.5 After each calibration standard has been run and the instrument has calculated the calibration curve, verify that the calibration is linear to an r^2 of >0.995. All points on the calibration line must fall within 15% of the line, with the exception of the lowest point, which must not deviate from the line by more than 25%. If these criteria are not met, abort the run and re-run the calibration. If the calibration continues to fail, remake the working standards, repeat the calibration, and continue.

4.4.6 If the absorbance of the sample is higher than the highest calibration standard, reduce the volume appropriately to not less than 5 µL. If the absorbance is still higher than the highest calibration standard, dilute the digestate in a larger acid cleaned vial. If the absorbance of most of the samples in the batch exceeds the highest calibration point, the calibration may be extended by running a higher calibration standard. The analyzer is linear to the range of a 50 µg/L standard. If the result of the 50 µg/L standard is within 10% of the original calibration, the system may be considered linear in that range and the data may be calculated using the original calibration.

4.4.7 Clean the analyzer by running 2-4 samples of 3% HNO_3, then deionized water (DI H_2O) through the machine at the end of each day.

4.5 Instrument Maintenance

4.5.1 The instrument is maintained by the analyst, with the assistance of service personnel at Thermo-Separation Products.

The following items are checked daily and changed weekly (under constant use):

- soda lime
- reagents (stannous chloride, 3% HNO_3, and rinse water)

The following items are checked weekly and changed bimonthly (under constant use):

- carbon trap
- filters

The following items are checked weekly and changed as needed:

- sample injection syringe
- tubing
- connectors
- lamp

4.5.2 The autosampler arm should be cleaned and lubricated bimonthly.

5.0 DATA ANALYSIS AND CALCULATIONS

5.1 The computer program calculates the concentration of Hg in the sample from either peak height or area, which is determined in the method file, by the following equations:

$[Hg] = ((PH_s * RF_1) + Rf_0) * V_d / W_d / 0.001*DF$

where:
- [Hg] = Mercury concentration (µg/g dry wt)
- Ph_s = Sample peak height or peak area
- RF_1 = Response Factor 1 (slope of the regression line in µg/area or height)
- Rf_o = Response Factor (y-intercept)
- V_d = Digested volume (mL)
- W_d = Digested weight (g)
- DF = Dilution Factor

5.2 Data Generation, Review, and Archiving and Software Maintenance

5.2.1 Instrument-generated data are checked using Excel software. All data points are recalculated from the raw peak area and compared to the instrument-generated value. The final data report is rechecked by the QA Officer during the data verification process.

5.2.2 Software is maintained by the analyst with the help of service personnel at Thermo-Separation Products. Backup instrument software is maintained on diskette. Data reduction is done using Excel software purchased and maintained under Battelle's software licensing agreement. Backup copies of all data are maintained on the mercury lab's shared drive, the m-drive.

5.2.3 Electronic data are backed up monthly and also maintained on the analyzer for 1 year. Electronic copies of the data summaries are maintained on the m-drive for two years and then either deleted, forwarded to the client, or archived on diskette as required by the client. Hard copies of all data are maintained in the central file system for at least 10 years.

5.3 Quality Assurance Data Reporting

5.3.1 Matrix Spike Recoveries

Matrix spike recoveries are reported as percent recovery:

% Recovery = 100* [spiked sample] – [sample only] / [spike added]

When matrix spike/matrix spike duplicate samples are analyzed, the relative percent difference (RPD) between the duplicates should be calculated:

RPD= (% recovery MS - % recovery MSD) / [(%Recovery MS + %Recovery MSD) / 2]

5.3.3 Analytical Replicates

Analytical precision is reported by calculating the relative percent difference (RPD) between analytical duplicates:

RPD = ([Rep 1] – [Rep 2]) / (([Rep 1] + [Rep 2]) / 2) * 100

Analytical precision is reported by calculating the relative standard deviation (RSD) between analytical replicates:

$$RSD = \frac{\text{standard deviation}_{(n-1)} \text{ of all replicates}}{\text{average of all replicates}} * 100$$

5.3.3 Laboratory Control Samples and Certified Reference Materials

Analytical accuracy is reported by calculating the percent recovery of laboratory control samples (LCS) and/or certified or standard reference materials (CRM or SRM) in comparison to the known concentration and/or certified value.

% Recovery = 100* [observed concentration] / [known concentration]

6.0 QUALITY CONTROL

6.1 One method blank, a sample duplicate, and matrix spike (on a representative sample, to check for matrix interference) should be analyzed per batch of samples or as specified by the customer. A method blank consists of all reagents used in the digestion procedure and it is digested and analyzed as a sample.

6.2 One standard reference material (SRM) should be digested and analyzed with each batch of samples or as specified by the customer, following step 4.4.4.

6.3 An initial calibration verification and continuing calibration verifications should be run every ten samples and must be within 15% of the original calibration. If this check fails, run a duplicate and if that fails rerun the calibration curve and all samples analyzed after the last passing calibration check.

6.4 Data quality objectives, acceptance limits, and corrective actions are outlined in the Table 1 below.

6.5 Method detection limits (MDLs) for total mercury are determined by two methods:

6.5.1 To determine general matrix specific MDLs: Analyze 7 replicates of a low level sample (clean sand for sediment or phytoplankton for tissue). Take the standard deviation $_{(n-1)}$ of the 7 replicates and multiply it by the Student's T-value for 7 replicates $_{(n-1)}$ as outlined in MSL-Q-007.

6.5.2 In cases when an empirically derived MDL is not practical (when a clean enough sample can not be found to produce a meaningful MDL for a specific matrix), a theoretical MDL may be calculated. To determine a theoretical sample-specific MDL: Analyze 7 replicates of a low-level liquid standard (0.5 µg/L). Take the standard deviation $_{(n-1)}$ of the 7 replicates and multiply it by the

Student's T-value for 7 replicates $_{(n-1)}$ to derive an instrument detection limit (IDL). Put the IDL through the sample concentration calculation (divide by volume analyzed and then multiply by total sample volume and divide by total sample mass).

Battelle MSL does not routinely employ reporting limits (RL). It is MSL policy to report all values detected above the empirically determined MDL (or calculated MDL in the case of tissues or sediments when no empirical MDL can realistically be determined). If a client specifically requests that data be reported below the MDL, the affected data should be flagged as detected below the detection limit according to the client's requested flagging convention.

Table 1.. Data Quality Objectives

QC Sample Type	Frequency	Acceptance Limit	Corrective Action
Laboratory Method Blank	1 per batch of 20 or fewer	<5 times the MDL	Reanalyze. If confirmed and all samples are >10 times the blank, no corrective action is required. If samples are <10 times the blank, the batch must be redigested.
Replicate Precision	1 per batch of 20 or fewer	25% for analytes >3 times the MDL No more than 35% of all RPDs can be > 25%[c]	Reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple DQO's requires redigestion and reanalysis of batch.
Certified Reference Material (CRM) or Standard Reference Material (SRM)	1 per batch of 20 or fewer	75-125% of certified value	Reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple DQOs requires redigestion and reanalysis of batch.
Matrix Spike (MS)	1 per batch of 20 or fewer	75-125% recovery	Reanalyze. Failure to meet criteria shall be reported in Data Summary. Failure of multiple DQOs requires redigestion and reanalysis of batch.
Initial and Continuing Calibration Verification	every 10 samples	<10% of initial calibration	Reanalyze. If subsequent ICV or CCV still fails, rerun the calibration curve and all samples analyzed after the last passing calibration check.

7.0 SAFETY

All analysts following this procedure should be aware of routine laboratory safety concerns, including the following:

- Protective clothing and eyeglasses should be worn when appropriate.
- Proper care must be exercised when handling samples.

8.0 TRAINING REQUIREMENTS

All staff performing total mercury in tissues and sediments must first read this procedure and then demonstrate proficiency in the process prior to performing any project work. Proficiency may be demonstrated and documented through successful analysis of certified reference materials, performance evaluation (PE) samples, or matrix spike/matrix spike duplicates. Documentation of training will be recorded on a training form obtained from MSL-A-006, Marine Sciences Laboratory Training.

9.0 REFERENCES

Thermo Separation Products HgTalk Mercury Analysis Software

Thermo Separation Products Operator's Manuals for Mercury Vapor Generator and Detector

MSL-A-006, Marine Sciences Laboratory Training.

ATTACHMENT 1
Example Spreadsheet

PROJECT ID: Spreadsheet Examples

ANALYSIS: TSP HG ANALYSER
ANALYSIS DATE:
MATRIX: Sediment/Tissue

ANALYST: Deuth
FILE #: ####

VIAL #	FILE #	SAMPLE ID	DIGEST WT g	DIGEST VOL ml	ANALYZ VOL ml	TUBE VOL ml	DILUT FACTOR	CALC VOL ml
1		0						
2		0.5 µg/l						
3		1.25 µg/l						
4		5.0 µg/l						
5		7.50 µg/l						
6		12.50 µg/l						
7		HNO3 Blk						
8		ICV Std #						
9	100Test	Blank	0.250	23.091	3.000	6	2	46.18
10	100Test	LCS 1	0.250	22.768	3.000	6	2	45.54
11	100Test	MESS-2	0.255	22.905	3.000	6	2	45.81
12	100Test	PACS-1	0.200	23.904	0.100	6.1	61	1458.14
13	100Test	1 R1	0.271	22.929	3.000	6	2	45.86
14	100Test	1R2	0.250	22.922	3.000	6	2	45.84
15	100Test	2	0.278	22.866	3.000	6	2	45.73
16	100Test	3	0.247	22.946	1.000	7	7	160.62
17	100Test	4	0.259	23.017	1.000	7	7	161.12
18	100Test	4 MS1	0.268	23.024	0.500	6.5	13	299.31
19	100Test	4 MSD1	0.258	22.942	0.500	6.5	13	298.25
20	100Test	CCV Std #						

ATTACHMENT 2
EXAMPLE SEQUENCE FILE

File Header Edit Global Change Validate

OVR	SDG	Vial #	Vial #	Phase	Type	Level	Amount	Dilu.Fact
1		2/1	0	liquid	blank	0	1	1
2		2/2	0.5 µg/l	liquid	standard	1	1	1
3		2/3	1.25 µg/l	liquid	standard	2	1	1
4		2/4	5.0 µg/l	liquid	standard	3	1	1
5		2/5	7.50 µg/l	liquid	standard	4	1	1
6		2/6	12.50 µg/l	liquid	standard	5	1	1
7		2/7	HNO3 Blk	solid	sample		1	1
8		2/8	ICV Std #	liquid	sample		1	1
9	100Test	2/9	Blank	solid	sample		0.250	46.182
10	100Test	2/10	LCS 1	solid	sample		0.250	45.536
11	100Test	2/11	MESS-2	solid	sample		0.255	45.810
12	100Test	2/12	PACS-1	solid	sample		0.200	1458.144
13	100Test	2/13	1 R1	solid	sample		0.271	45.858
14	100Test	2/14	1 R2	solid	sample		0.250	45.844
15	100Test	2/15	2	solid	sample		0.278	45.732
16	100Test	2/16	3	solid	sample		0.247	160.622
17	100Test	2/17	4	solid	sample		0.259	161.119
18	100Test	2/18	4 MS1	solid	sample		0.268	299.312
19	100Test	2/19	4 MSD1	solid	sample		0.258	298.246
20	100Test	2/20	CCV Std #	liquid	sample		1	1

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Marine Sciences Laboratory EFFECTIVE DATE: 4-10-02

Battelle Pacific Northwest National Laboratories
Marine Sciences Laboratory

STANDARD OPERATING PROCEDURE
MSL-A-002-03

SAMPLE CHAIN-OF-CUSTODY

Approvals:		
AUTHOR: Deborah Coffey	Deborah Coffey	4-10-02
	Signature	Date
TECHNICAL REVIEWER: Carolynn Suslick	[illegible]	4-10-02
	Signature	Date
QA OFFICER: Deborah Coffey	Deborah Coffey	4-10-02
	Signature	Date
TECHNICAL GROUP MANAGER: Eric Crecelius	Eric Crecelius	4-10-02
	Signature	Date

SAMPLE CHAIN-OF-CUSTODY

1.0 SCOPE AND APPLICATION

This procedure defines the methods for establishing the traceability of samples transferred to the Battelle Marine Science Laboratory (MSL) for chemical and/or biological testing. This process ensures the integrity of the samples from the time of collection through sample disposal. The sequential custody of samples will be documented using this procedure. Each custodian of the samples shall comply with the procedures described below.

2.0 DEFINITIONS

- **Custody** - Having control of the sample in one or more of the following manners: 1) physical possession; 2) in person's view after taking possession; 3) secured by a person in a manner that prevents tampering of sample; and/or 4) secured by a person in an area restricted to authorized personnel.

- **Sample Custodian** - The person assigned, at a given field site, laboratory, or testing facility, for having responsibility for custody of the sample.

- **LRB** - Laboratory Record Book

- **CoC** - Chain of Custody

3.0 RESPONSIBLE STAFF

Marine Sciences Laboratory (MSL) Staff as Sample Custodian or as Sample Recipient or as MSL Contact
Project Manager or Task Leader
MSL Manager
MSL Quality Assurance Officer or Representative

4.0 PROCEDURE

4.1 Custody Procedures in the Field or Laboratory

4.1.1 The sample custodian may be a member of the sampling crew or a person that works with those who are collecting the samples. The sample custodian ensures that sample labels are filled out and affixed to the appropriate sample containers before or at the time of sample collection.

Information on the sample labels may include, but not be limited to, a code number identifying the sample, date, time, and location of sample collection, and name of sample collector.

4.1.2 Once the samples are collected, the sample custodian records pertinent sample collection information on required raw data documentation (i.e., sample log, LRB, etc.). Information may include, but not be limited to, a code number identifying the sample, date, time, and location of sample collection, and name of sample collector.

Record in permanent ink all pertinent information about each sample on a Chain-of-Custody Form (Attachment 1 or 2). Press hard when making entries and assure transfer to carbon copies. Multiple samples collected on the same date may be recorded on one Chain-of-Custody Form, provided each sample is identified individually on the form.

Note: **The Field Sample Chain of Custody (Attachment 1) is used primarily when transferring samples from the field to the MSL for processing. The Sample Custody Record (Attachment 2) is used when transferring samples from the field or the MSL to another laboratory or testing facility. For the purpose of this SOP, the term Chain-of-Custody Form can mean either of the two forms.**

4.1.3 If required by a project-specific protocol, the sample custodian attaches custody seals to the samples or to the shipping container (e.g., ice chest) immediately on sample collection. The seal is attached in such a way that the sample cannot be opened without breaking the seal.

4.1.4 If there are special storage requirements (i.e., temperature requirements), the sample custodian ensures that samples are immediately stored using the required method and appropriate containers.

4.1.5 The sample custodian is responsible for the samples during delivery to the MSL, laboratory or testing facility until custody of the samples can be transferred to the sample recipient or until release of the samples during shipment (e.g., if samples have to be shipped via overnight carrier, etc.). If custody of the samples cannot be transferred to the sample recipient or shipped on the same day as sample collection, the samples must be stored in a locked or secured storage area until the transfer can be made.

Note: **Chain-of-Custody Forms shall remain with samples during transfer.**

4.2 Transferring Custody of Samples to a Laboratory or Testing Facility

4.2.1 Upon arrival at the laboratory or testing facility or just prior to releasing samples for shipment, the sample custodian examines the sample container(s) to ensure that the sample seals are intact and the sample containers have not been damaged.

4.2.2 The sample custodian relinquishes custody by signing, dating, and noting the time in the "Relinquished By" space on the Chain-of-Custody Form. The sample custodian tears off the bottom copy (pink) of the Chain-of-Custody Form and retains it for filing with project files.

4.2.3 The sample recipient takes custody of the samples by signing, dating, and noting the time in the "Received By" space on the Chain-of-Custody Form. The sample recipient now becomes the laboratory sample custodian, completing the transfer of sample custody. The contents of the shipping container must be checked against the information on the chain-of-custody form for anomalies. If any discrepancies are noted, or if laboratory acceptance criteria or project-specific criteria are not met, the laboratory must contact the client's designated point of contact for resolution of the problem. The discrepancy, its resolution, and the identity of the person contacted must be documented in the project file. If any seals have been broken and/or the sample containers are damaged, the sample recipient records the condition of the seals and containers in the remarks section of the Chain-of-Custody Form.

4.2.4 The Chain-of-Custody Forms travel with the samples during the transfer, and are filed in the laboratory or testing facility's project files.

4.3 Internal Chain of Custody

MSL does not routinely invoke a formal internal chain of custody process. Access to the building is limited by requiring all staff members to have electronic access cards to enter the building (refer to MSL-A-011, Marine Sciences Laboratory Access Control.) Visitors are issued daily badges. After hours site access is maintained by a gated fence to the grounds and the presence of a security guard. Non-analytical staff are not encouraged to be in areas when they have no reason to be there.

The laboratories are physically located in close proximity to one another and samples are within the physical control of the analysts during digestion and analysis activities. Samples are received in the shipping and receiving area, and logged in per procedure MSL-A-001, Sample Log-In Procedure, and stored until digestion (if required) and analysis. MSL does not store digestate for re-analysis. Instead, if a sample requires re-analysis it is digested from an archived sample. Access to sample archive refrigerators and freezers is restricted.

A Log-in Checklist (see Appendix of MSL-A-001) is used to document sample sample receipt activities, verification of field sample preservation, sample filtration and preservation when required, and to document any deviations related to sample receipt and sample log-in.

MSL documentation provides the location of the sample post-receipt on the Sample Log-In Form. Digestion sheets provide a record of sample digestion dates. Analysis times are documented on raw data print outs. Sample disposition is determined by the client and is documented on the Log-In Checklist in the section completed by the Project Manager. Sample disposition processes are documented in MSL-I-026, Use of Laboratory Refrigerators and Freezers. When an internal chain of custody report is desired, it can be generated from data sheets in the sample analysis data file, and verified against the data file documentation by the Project Manager and MSL QA Officer.

4.4 Evidentiary/Legal Chain of Custody

In a few cases, clients request samples to be tracked under evidentiary/legal chain of custody requirements. In these cases MSL is prepared to establish an intact, continuous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates, accounting for all time periods associated with sample receipt, processing, analysis and storage and disposal. When evidentiary/legal chain of custody is requested, MSL documents all sample fates in a summary traffic report for the client based on objective evidence maintained during the sample processing. Objective evidence will be defined as all information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis, reporting, archiving, and disposal. Including signatures of all individuals who physical handle individual samples.

When evidentiary/legal chain of custody is required, the assigned project manager will discuss the following requirements with the client to determine which items are required and to ensure that all relevant items are addressed because different programs have different requirements and to assist in project planning.

1. The point at which evidentiary/legal chain of custody is initiated and whose responsibility it will be must be defined.

2. Determine if samples will be shipped in individual sample containers with custody seals intact.

3. Determine if samples will be shipped in coolers with custody seals intact.

4. Determine if the chain of custody forms will remain with the samples during transport or shipment.

5. Determine if the tracking records for legal COC will include the time of day and calendar date of each transfer or handling procedure.

6. Is the expectation that the laboratory will retain the receipts of packages sent by common carrier as a part of the permanent chain-of-custody procedure?

7. Determine if the desired level of evidentiary/legal chain of custody includes the requirements that laboratory personnel: (1) are responsible for the care and custody of the sample and (2) prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed or the sample is disposed

MSL is not always able to control steps 1-5 above, when MSL is not responsible for sample collection, labeling, preservation, handling, and shipment.

4.5 Subdividing Samples

Once at the MSL laboratory or testing facility, if samples have to be subdivided and submitted to a subcontractor laboratory, this information will be noted on the original Chain-of-Custody Form (from sample collection), and a new Chain-of-Custody Form is initiated. With each transaction, the sample custodian relinquishes custody to the sample recipient, who then becomes the next sample custodian. (See Sections 4.2.2 through 4.2.4 above.) The requirements for chain of custody and sample disposition will be noted on the Chain-of-Custody form.

4.6 Disposal of Samples

4.6.1 When samples are disposed of by the subcontractor laboratory:

- If the subcontractor laboratory or testing facility is responsible for disposing of the samples, the subcontractor is asked to notify the MSL Project Manager before final disposition. The MSL Contact will notify the originator that the samples are scheduled to be destroyed, or will define customer requirements for an extended period of storage.

- After destruction of samples, the subcontractor laboratory or testing facility is asked to return a copy of the Chain-of-Custody Form to the MSL Contact for placement in project files. The originator may be forwarded a copy of the final Chain-of Custody documentation if requested.

- The MSL Contact records the date of receipt on the Chain-of-Custody Form in the "Received by" section of the form space and indicates the samples were destroyed ending the chain of possession.

4.6.2 When samples are disposed of by the Marine Sciences Laboratory (MSL):

- If the laboratory or testing facility is not responsible for disposal of the samples, MSL personnel will obtain custody of the samples from the subcontractor laboratory or testing facility along with the Chain-of-Custody Form.

  For returned samples or samples that have never left MSL custody, the MSL Contact will notify the originator that the samples are scheduled to be destroyed, or will define customer requirements for an extended period of storage.

  If extended storage is not requested, then MSL will dispose of the samples following the guidelines specified in the Pacific Northwest National Laboratory's (PNNL's) Standards-Based Management System (SBMS). This system provides a framework for logging in reagents, chemicals and solutions into the associated Chemical Management System (CMS). This system provides the PNNL Laboratory with the policies and procedures regarding tracking and inventory, storage and disposal of completed samples and analytical wastes, as well as chemical use and disposal. The CMS is used to provide an up-to-date inventory to facilitate emergency response, monitor the location of various classes of materials and identify situations where acceptable limits for the building/facility determined by the assigned chemical hazard group and fire zone might be exceeded before a violation occurs.

- After destruction of samples, MSL personnel responsible for sample destruction returns a copy of the Chain-of-Custody Form to the MSL Contact and the Sample Disposal Log Book entry is updated.

- The MSL Contact records the date of receipt on the Chain-of-Custody Form in the "Received by" space next to the Sample Custodian's signature and indicates the samples were destroyed ending the chain of possession.

4.6.3 When samples are returned to the customer for disposal:

- Samples may be returned to the customer (or the sampling site) by customer request. Samples are shipped to meet Department of Transportation regulations Generally, the samples are shipped in the same way that they were initially shipped to MSL. Sample disposition should be documented in the central file of each project.

4.5.4 The MSL Contact shall ensure that completed Chain-of-Custody Forms are filed in the appropriate project files. The originator may be forwarded a copy of the final Chain-of Custody documentation if requested.

5.0 DATA ANALYSIS AND CALCULATIONS

There are no calculations applicable to this procedure.

6.0 QUALITY CONTROL

It is the responsibility of each individual taking or relinquishing custody of the samples to ensure that Chain-of-Custody Forms are filled out accurately and completely for each transaction, and that the forms are filed in the appropriate project files.

If the Chain of Custody is broken at any time when the sample is in the control of MSL, this deviation must be documented in the data report narrative.

7.0 SAFETY

Not applicable.

8.0 TRAINING REQUIREMENTS

All staff responsible for sample custody (i.e., sample relinquisher or sample recipient) shall first read this procedure and document the training as a completed reading assignment on an Individual Training Assignment Form or a Group Training Documentation Form as described in MSL-A-006, Marine Sciences Laboratory Training.

9.0 REFERENCES

MSL-A-001	Sample Log-In Procedure
MSL-A-006	Marine Sciences Laboratory Training
MSL-A-011	Marine Sciences Laboratory Access Control
MSL-D-004	Data Reporting, Reduction, Back Up, and Archiving
MSL-I-026	Use of Laboratory Refrigerators and Freezer

Attachment 1

Battelle Marine Sciences Laboratory 1529 W. Sequim Bay Rd. Sequim, WA 98382	**EXAMPLE FIELD SAMPLING CHAIN OF CUSTODY**	Page ____ of ____

Shipped To:

Company:

Address:

Method of Shipment:

Shipped From: By:

Telephone:

SPECIAL INSTRUCTIONS:

Container No.:

Sampling Location:

Samples Collected By: Date:

Remarks:

SAMPLE IDENTIFCATION

Relinquished by	Date/Time	Received by	Date/Time
Relinquished by	Date/Time	Received by	Date/Time
Relinquished by	Date/Time	Received by	Date/Time

ATTACHMENT 2

EXAMPLE
SAMPLE CUSTODY RECORD

Date ________
Page of

Battelle
Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, Washington 98362

Project No.:

Project Name:

Project Manager:

Phone:

Laboratory:

Address:

Attn:

Testing Parameters

Lab No.	Sample No.	Collection Date	Matrix			Number of Containers	Observations, Instructions
							total # of containers

Relinquished by:			Received by:			Shipment Method:
Signature	Date	Time	Signature	Date	Time	**Special Requirements or Conditions:**
Printed Name	Company		Printed Name	Company		**Sample Disposition:**
Relinquished by:			Received by:			Distribution: 1. 2 copies to the Laboratory 2. 1 copy project manager 3. Return completed original to Battelle Marine Sciences Laboratory
Signature	Date	Time	Signature	Date	Time	
Printed Name	Company		Printed Name	Company		

SAMPLE CUSTODY RECORD
(SOP# MSL-A-001 & MSL-A-002)

Date: ____________

Battelle
. . . Putting Technology To Work
Pacific Northwest Division
Marine Sciences Laboratory
1529 West Sequim Bay Road
Sequim, Washington 98382

Project Name: ____________
Project Manager: ____________
Phone Number: ____________

Shipment Method: ____________
Preservation: ____________

Line	Field Sample ID	Collection Date/Time	Matrix	No. of Containers	Test Parameters					Laboratory ID	Observations/Comments
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											

Relinquished By: Company:
Signature/Printed Name Date/Time

Received By: Company:
Signature/Printed Name Date/Time

Relinquished By: Company:
Signature/Printed Name Date/Time

Received By: Company:
Signature/Printed Name Date/Time

Rev. 020102COC

Distribution: White - Laboratory, Project Files Yellow - Laboratory, Client Pink - Sampler

Weatherproof Laser Labels

Use template for 5164™

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

Battelle Marine Science Laboratory
Container Specially Cleaned for Trace Metals
Date:

Sample ID:	**Date:**
Lab ID:	**Time:**
Site Description:	**Collected by:**
Parameters:	**Preservative:**

SOP #BR-0011

Determination of Methyl Mercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation, and CVAFS Detection: BRL Procedure for EPA Method 1630

Brooks Rand, LLC

Revision 008
Written 1/90
Revised 4/7/03

Reviewed ____________________

____

____

____

____________________ President — 4/17/03 Date

____________________ QA Manager — 4/9/03 Date

____________________ Senior Scientist — 4/8/03 Date

____________________ Scientist (if applicable) — 4/9/03 Date

Determination of Methyl Mercury by Aqueous Phase Ethylation, Trapping Pre-Collection, Isothermal GC Separation, and CVAFS Detection: BRL Procedure for EPA Method 1630

1. SCOPE AND APPLICATION

1.1. Method BR-0011 is the performance based procedure followed at BRL as EPA Draft Method 1630. Unless specifically stated otherwise in this document, all apparatus, materials, reagents, standards and procedures as stated in EPA Method 1630 are used at BRL.

NOTE: EPA Draft Method 1630 is for the determination of methyl mercury only in filtered and unfiltered aqueous samples. Brooks Rand Method BR-0011 is additionally for the determination of methyl mercury in sediment and biota. Brooks Rand has developed specific sample preparation methods for these matrices. With the exception of the maximum volumes analyzed, the procedures followed for the analysis of sediment and biota preparations are identical to the procedures followed for aqueous preparations.

2. SUMMARY OF METHOD

2.1. Prior to instrumental analysis, the aqueous and sedimentary samples are prepared by distillation at 145°C under N_2 according to the procedure discussed in EPA Draft Method 1630, section 11. Biota samples are prepared by alkaline digestion in 25%KOH in Methanol and oven digesion at 65°C.

2.2. Mono-methylmercury (MMHg) is determined by an improved method (Liang, Bloom, and Horvat 1994). The MMHg is first ethylated with sodium tetraethylborate ($NaBEt_4$) and collected by purging with dry, Hg free nitrogen onto a quartz tube filled with either Carbotrap™ or Tenax. The ethyl mercury derivatives are then thermally desorbed and transferred to a GC column held in an oven at 105° C, which separate the species chromatographically by mass. The ethylated Hg compounds are pyrolized at 900° C to Hg(0), then quantified by a cold vapor atomic fluorescence spectrophotometer (CVAFS). This method can be applied for the determination of MMHg in a variety of sample matrices and has been demonstrated as being very sensitive, precise, and accurate. Very good results were obtained for the determination of MMHg in standard and certified reference materials and numerous intercalibration samples (Liang, Bloom, and Horvat 1994).

3. INTERFERENCES

3.1. If properly applied, the distillation procedure will remove most to all significant interferences. However, the concentration of HCl in the solution will affect the distillation of methyl mercury from the solution. Too little HCl will cause the distillation of methyl mercury to not be quantitative while too much HCl will cause the co-distillation of HCl fumes, which interfere with the ethylation process. EPA Method 1630 dictates that fresh water samples must be preserved with between 0.3% to 0.5% (v/v) 11.6 M HCl (BRL preserves fresh water samples with 0.4% (v/v) 11.6 M HCl) and that salt water samples must be preserved with between 0.1% to 0.2% (v/v) 9 M H_2SO_4 (BRL preserves salt water samples with 0.2% (v/v) 9 M H_2SO_4).

3.2. Samples must not be preserved with nitric acid as it may cause partial decomposition of the analyte during distillation.

3.3. Positive artifact is possible with the distillation of samples that are high in inorganic mercury. Ambient organic matter may methylate 0.01% to 0.05% of the ambient inorganic mercury during distillation. In inorganic mercury contaminated waters this can significantly affect the results for methyl mercury. Solvent extraction may be preferable to distillation in samples that are high in divalent mercury (Hg(II)).

3.4. Refer to EPA Method 1630, Section 4.0 for a detailed account of possible contamination and interference to the analysis and how these are avoided or minimized at BRL.

4. APPARATUS AND MATERIALS USED AT BRL

4.1. Refer to EPA Method 1630, Section 6.0 for a list of materials used in the method employed at BRL.

4.2. Detailed instructions for the decontamination of bottles and other equipment are described in BRL SOPs BR-0400 (Decontamination of Sampling Containers), BR-0401 (Mercury Decontamination of Silicon Tubing), BR-0402 (Mercury Decontamination of Glassware), BR-0403 (Mercury Decontamination of Miscellaneous and Small Items), and BR-0404 (Preventing Mercury Contamination of Samples).

4.3. Specific equipment used at BRL is listed below. Any modifications to EPA Method 1630 are described and explained.

4.3.1. Atomic fluorescence spectrophotometer (BRL part #AF-03): CVAFS systems are built by Brooks Rand, LLC (BRL Model III). Refer to the "Brooks Rand, LLC Model III Operations Manual" for instrument operating instructions.

4.3.2. Data Acquisition : Integration software (BRL Mercury Guru Version 2.0 or later) with PC for peak area measurements. Alternatively a chart recorder (Yokogawa 3021) for peak height measurements may be used. The installation and use of the Guru software is described in the "Brooks Rand, LLC Model III Operations Manual."

4.3.3. Reaction and purge vessels (BRL part #AF-32): A 150 mL flat bottom bottle with 24/40 tapered fitting is used as the reaction vessel, in conjunction with a special 4-way valve sparging-tube cap-assembly. This valve assembly allows the prepared sample to react with the ethylating reagent without bubbling, and then to be purged onto the trapping column. Finally, the valve assembly allows the prepared sample to be bypassed so that water vapor adsorbed onto the column may be evaporated by the direct flow of dry carrier gas.

4.3.4. Trapping column (BRL part #AF-21): Tenax traps used for the collection of purged organomercury species measuring 10 cm long of 6.4 mm outside diameter x 4.0 mm inside diameter. Figure 1 also shows the connection of the column and a reaction vessel.

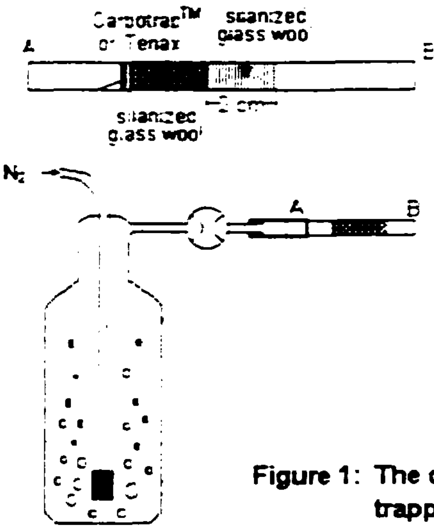

Figure 1: The construction of trapping column and its connection with a reaction vessel

4.3.5. Guard Column: If a Tenax TA trap is to be used for collecting purged species, a pre-GC column OV-3 trap must also be used. This guard column is placed between the Tenax trap and the GC column and serves to inhibit Tenax material from entering the GC column. The guard column consists of approximately 70 mg of OV-3 3% packed in the same type of quartz tube used for the trapping column, between quartz wool plugs. The guard column is initially conditioned by heating twice for 30 seconds, allowing the trap to cool between heatings. The guard column should be blanked daily with the trapping columns before analysis. Guard columns should be replaced routinely at least every four (4) months or when a problem arises.

4.3.6. Isothermal gas chromatography system: Consisting of GC column (BRL part #AF-34), GC oven (BRL part #AF-33), pyrolitic column (AF-35) and temperature controller for GC oven (BRL part #AF-36). For a diagram of the system see Figure 2. Under a 30 mL/min flow of high purity helium, organomercury species desorbed from a trapping column are carried by gas passing through the GC column, held at 105° C in a cylindrical oven, and eluted. Separated species are decomposed in a thermal decomposition tube and finally detected by CVAFS.

5. STANDARDS AND REAGENTS

5.1. Refer to EPA Method 1630, Section 7.0 for a list of standards and reagents employed at BRL.

5.2. Water: 18 megohm ultra-pure deionized water starting from a pre-purified (distilled, R.O., etc.) source. As a final mercury and organic removal step, the activated carbon cartridge on the 18 megohm system is placed between the final ion exchange bed and the 0.2 μM filter. Deionized water is monitored on a daily basis for conductivity with a VWR "Pure H_2O Tester" conductivity meter. The deionized water must have a conductivity ≤ 1.0 μS/cm to be used. Double Deionized Water (DDW) from a Millipore System is used throughout.

5.3. MMHg Standard solutions

a) Standard stock solution: 1 mg/mL MMHg is purchased from a known, accredited vendor. This standard is used to prepare the calibration standards. The standard used to prepare the independent check standard is purchased from a separate known, accredited vendor than the calibration standard. The current concentration of the independent check standard stock solution is 0.88M MMHg. A portion of this solution is diluted with DDW to a concentration of 1 mg/mL MMHg and stored in a 125 mL fluoropolymer bottle from 0-4°C.

b) Intermediate stock solution: 1 μg/mL MMHg. Dilute 0.10 mL of 1 mg/mL stock solution to 100 mL with DDW. This solution expires after one month.

c) Working standard: 1 ng/mL MMHg. Dilute 0.10 mL of 1 μg/mL intermediate stock solution to 100 mL with DDW. This solution expires daily.

5.4. Sodium tetraethylborate ($NaBEt_4$) solution: Dissolve 1.0 g of $NaBEt_4$ (stored in freezer) in 100 mL of 2.0% KOH solution that has been chilled to 0° C to produce a 1.0% working solution. (Note: The solution may be sparged with N_2 overnight to reduce excess mercury if necessary.) This solution is decanted into individual 5.0 mL fluoropolymer vials. This reagent is stored at < -10° C and is defrosted prior to use. The solution expires daily upon unfreezing. A new batch of the ethylating reagent should be made as soon as there are any doubts about its quality (i.e. low recovery of matrix spikes). $NaBEt_4$ solids and solutions must not be used if they have become yellow.

5.5. Sodium acetate buffer: A 2M acetate buffer is prepared by dissolving 272 g of reagent grade sodium acetate and 118 mL of glacial acetic acid in DDW to a final volume of 1 L. This solution is purified of trace mercury by the addition of 5 g of 1 N HCl-rinsed sulfhydoxyl chelating resin (Sumitomo Q-10R) to the bottle and agitation. The solution is stored in a Fluoropolymer bottle.

5.6. Methanolic potassium hydroxide solution: Dissolve 250 g of reagent grade KOH pellets in high purity methanol to a final volume of 1 L. The solution is stored in a Fluoropolymer bottle.

5.7. Gases: Helium used as a GC carrier gas is ultra high-purity grade. Nitrogen used as a purge gas for sweeping derivatives from a bubbler is plumbed from cryogenic bleed-off. Both are passed through a gold-coated sand trap to remove traces of mercury prior to use.

5.8. 20 % KCl 0.2 % L-Cysteine solution: Dissolve 10.0 g KCl and 0.1 g L-Cysteine in 50 mL of DDW. This solution must be discarded and replaced every 6 months or if crystals begin to form.

5.9. 9 M H_2SO_4: Mix equal parts DDW and pre-analyzed, concentrated H_2SO_4. Introduce the reagents slowly as this procedure generates a great deal of heat. Allow to cool completely before capping tightly.

5.10. 0.05 % $NH_2OH{\bullet}HCl$: Dilute 400 µL of 30 % $NH_2OH{\bullet}HCl$, used for Total Hg analysis, to 240 mL DDW. Discard and replace after one month.

5.11. Potassium Bromide/Sulfuric Acid solution: 18% (w/v) KBr + 5% (v/v) H_2SO_4. Dissolve 18.0 g of KBr into 100 mL of 5% H_2SO_4. This solution is stable for up to one year.

5.12. 1M Copper sulfate solution: Dissolve 24.97 g of copper(II) sulfate pentahydrate into 100 mL DDW. This solution is stable for up to one year.

5.13. Dichloromethane: Dichloromethane (HPLC Grade) may be purchased from an authorized vendor. The dichloromethane is purified of trace mercury by the addition of 10 g of 1 N HCl-rinsed sulfhydoxyl chelating resin (Sumitomo Q-10R) to the bottle and agitation.

6. SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1. Refer to EPA Method 1630, Section 8.0 and EPA Method 1669 (*Sampling Ambient Water for Determination of Trace Metals at EPA Water Quality Criteria Levels*) for a detailed description of sample collection, preservation, and storage methods.

7. SAMPLE PREPARATION

7.1. Refer to EPA Method 1630, Section 11.0 for a detailed description of the preparation of samples. Depending on the purposes and definitions of investigations of mercury biogeochemistry cycling, samples are prepared in the following methods prior to analysis.

7.2. Preparation of aqueous samples for MMHg analysis.

The following two isolation methods, distillation and solvent extraction, have been used in our labs for the determination of MMHg in aqueous samples. Good agreement was obtained in the comparison of the two methods for most water samples studied: for organic rich and/or high level sulfide containing samples, the distillation showed some advantages over the solvent extraction method with higher recoveries (85 ± 4%, Horvat, Bloom, and Liang, 1993). In addition, extraction consumes large quantities of organic solvent which can result in environmental contamination. Therefore, distillation is the preferred sample preparation method at BRL.

7.2.1. Distillation:

Reagents: 20% KCl in 0.2 % L-Cysteine, 9 M H_2SO_4, 0.05% $NH_2OH \cdot HCl$

Distillation devices: Vials and caps for distillation and distillate collection are made of Fluoropolymer obtained by Savillex Corporation, USA. Caps have 1/8" ports for friction fit 1/8" Fluoropolymer tubing. Instead of Fluoropolymer, a glass distillation still may also be used (Horvat and Stoeppler, 1988).

Distillation procedures: An aliquot of water sample, typically 45 mL, is transferred into a 60 mL Fluoropolymer vial (for high MMHg concentration samples, small sample size should be used, but bring the final volume to a known volume with DDW). Add 0.2 mL of the 20% KCl/0.2 % L-Cysteine solution and 0.5 mL of 9 M H_2SO_4. Start the distillation immediately after addition of reagents at a nitrogen flow rate of approximately 73 $mL \cdot min^{-1}$ (rotometer set at 10) and at a heating block temperature of 145° C. <u>Note</u>: The liquid nitrogen tank must be at least 1/8th full prior to beginning any distillation. This is judged by the gauge on top of the tank and <u>not</u> by the tank pressure. The distillate is collected in a 60 mL Fluoropolymer vial containing 5 mL of 0.05% $NH_2OH \cdot HCl$ in DDW, which is cooled in an ice-water bath. The distillation is finished when the final distillate volume is 45 mL, as measured against a reference vial. This typically takes from 2 to 3 hours. Bring the final volume of the receiving vial to the 58 mL mark with DDW. Depending on its MMHg concentration, transfer an aliquot of the distillate into the methylation reaction vessel for analysis as described in section 4. <u>Note</u>: Open the flow of nitrogen for up to one hour before beginning the distillation to purge the lines of room air and other possible contaminants.

7.2.2. Solvent extraction

Reagent: 30% KCl (saturated), methylene chloride (large blanks in MMHg determination occasionally result from this solvent. Therefore, different brands and lot numbers should be examined to minimize this contamination.)

Extraction procedure: An extraction procedure described by Bloom (1989) was used. Depending on its concentration, weigh an approximate volume of the sample acidified to a pH of 2-5, typically 50 mL into a 125 mL Fluoropolymer bottle. If a smaller sample size is used, bring the final volume to 50 mL with DDW. Add 5 mL of 30% KCl, and swirl the bottle to mix. Add 40 mL of methylene chloride. Shake the bottle for 1-2 h with a mechanical shaker to reach a distribution equilibrium of MMHg between aqueous and solvent phases, then allow the two phases to separate. Remove the upper phase (aqueous phase) by pipetting. Add about 50 mL of DDW to the methylene chloride, and place the uncapped bottle in a hot water bath at 60° C until all of the CH_2Cl_2 has boiled away. Be aware that the methylene chloride can boil suddenly in bursts, sending water and solvent into nearby bottles. Slowly bringing the bath up to temperature can hinder this effect. Watch the bottles to see if a steady boiling arises; if not, try rearranging the bottles (heat may be unevenly distributed on the bath floor if using an electric skillet). After all visible solvent has evaporated, continue heating for ten minutes. Then purge the water for 2-3 minutes at 250 mL·min^{-1} with Hg free N_2 to remove any residual solvent. The MMHg is transferred to the DDW matrix, which is ready for ethylation as described above.

7.3. Preparation of biological materials and sediments for MMHg.

7.3.1. Alkaline digestion: Weigh about 0.1 gram of biological material (wet, homogenous) into a 2.5 mL Fluoropolymer vial. Add 1.0 mL of 25% KOH methanol solution and cap the vial tightly. Digest the sample in an oven at 65° C for 3-4 hours. Avoid heating overnight, as recoveries drop sharply; recoveries may return to expected levels after sitting for several hours at room temperature. After digestion, bring the final volume to 2.5 mL with methanol prior to analysis. Analyze an appropriate aliquot, depending on the sample's concentration of MMHg and Hg(II). The day of analysis (if different from day of preparation), samples should be shaken thoroughly, heated in the oven at 65° C for 15-20 minutes and aliquots analyzed after samples have cooled and particulate settled. Alternatively, the digestion may be scaled up if larger volumes are required. One gram may be weighed into a 25.6 mL vial, and 10 mL of 25% KOH methanol solution added prior to digestion. Special care must be exhibited with biota certified reference materials as the fine particulate material can interfere with the analysis causing low recoveries. Sufficient time must be given for the fine particulate material to settle without allowing the CRM preparation to cool too much.

7.3.2. Distillation: Sediment samples should be distilled directly by weighing an appropriate amount, typically 1 gram, into a 25.6 mL Fluoropolymer vial and adding 15 mL of DDW. Distill as per the procedure mentioned above (7.1.1). If for biological samples, the MMHg concentration compared to Hg(II) is low, matrix interference on ethylation reaction caused by using large volumes of alkaline digestate will occur. This interference is avoided by distillation (Horvat, Bloom, and Liang, 1993). Distill after alkaline digestion by transferring 0.5-2.0 mL of alkaline digestate into a 25.6 mL vial, adding 15 mL DDW and following the procedure outlined in section 7.1.1.

7.3.3. Solvent extraction for sediments: Alternately, sediment samples may be extracted to avoid the potential for artifact formation of MMHg during distillation. Approximately 500 mg of sediment sample is accurately weighed into a clean glass vial with a Teflon lined screw cap. 5 mL of the potassium bromide/sulfuric acid solution (Section 5.11) and 1 mL of the 1M copper sulfate solution (Section 5.12) are added to the sample which is then allowed to leach for one hour. After leaching, 10 mL of dichloromethane are added using a volumetric glass pipette. The sample is intermittently shaken by hand over the next hour and then centrifuged for 30 minutes at 3000 rpm to assist in the separation of the aqueous layer from the organic layer. The sample is then passed through phase separating filter paper so that only the organic layer is collected. 2 mL of the extract are transferred to a clean, triple rinsed 58 mL Teflon vial that has been partially filled with approximately 45 mL of DDW. The sample is then heated to 45°C and purged with nitrogen for about 30 minutes until the the dichloromethane layer has evaporated off. The sample is then diluted to 58 mL with additional DDW. The sample is analyzed following the same procedure used for the analysis of water and sediment distillations.

7.4. Holding times for sample preparations.

7.4.1. Distillations: Water and sediment distillates are stable for up to 48 hours if stored at room temperature and in the dark. Distillates must not be refrigerated or frozen.

7.4.2. Extractions: Water and sediment extractions (once back extracted into water) are stable for up to 48 hours if stored at room temperature in the dark. The holding time limit for intermediate organic extraction (prior to back extraction) is currently being examined. Currently, Brooks Rand performs the entire extraction procedure within one day.

7.4.3. Digestions: Biological digestates are more stable than distillates and may be stored up to seven days prior to analysis.

8. INSTRUMENT CALIBRATION AND SAMPLE ANALYSIS

8.1. Refer to EPA Method 1630, Sections 10.0, 11.0, and 12.0 for a detailed description of the analysis of samples and the calculation of results.

8.2. Instrument Calibration: BRL follows EPA method 1630, Section 10.0 for the instrument calibration with the same exceptions as for sample analysis. Standards, typically 10, 50, 100, 250, 1000, and 2000 pg for MMHg are added into reaction vessels containing 50-75 mL of DDW and 200 µl of 2M acetate buffer.

8.3. Instrumental Analysis: BRL has adopted the following modifications.

For samples, add appropriate sample volumes plus DDW as necessary for a final bubbler volume of 50 to 75 mL (for sample preparation see Section 7 of this SOP). Some acid can be carried over during distillation: Adjust pH in the bubbler to approximately 6.2 by adding 20% KOH and or 1:1 HOAc solutions before adding buffer, if needed. (Solution should be clear or yellow in the presence of methyl red indicator.) Subsequent addition of buffer will bring the pH to the optimum range of 3.5-5.5. An aliquot of 50 µl (75 µl for biota samples) of $NaBEt_4$ is added, the 4-way valve-cap inserted and clamped, and the vessel swirled to wash any droplets back into solution. Allow the mixture to react without purging for 15 minutes. Place a trapping column in the orientation shown in Figure 1, and then purge with N_2 at a flow rate of 250 mL·min^{-1} (rotometer set at 35) for 15 minutes. The organomercury compounds are swept from solution and collected onto the trapping column. Then the valve is switched to pass dry gas over the column for 5 minutes, to remove water condensation from the trap. Biota samples should be allowed to react without purging for 20 minutes, purged with N_2 for 15 minutes and allowed to dry for 5 minutes. Afterwards, connect the trapping column in-line with the GC column as shown in Figure 2.

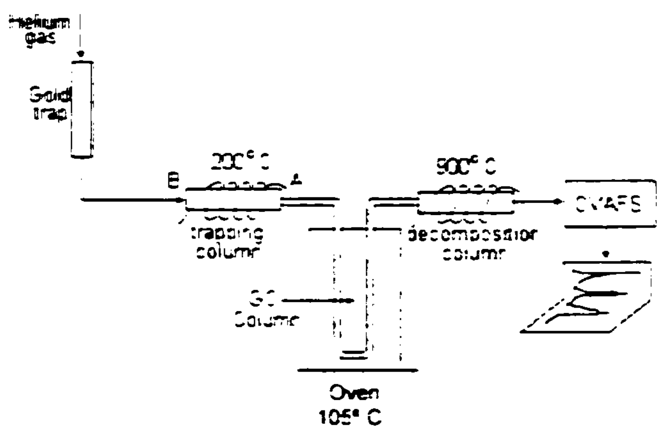

FIGURE 2: A schematic diagram of isothermal gas chromatograph system

When using a Tenax trap, special attention must be paid to the orientation of the trap. The trap is placed so that the end facing the bubbler output is now facing the GC column input to avoid the organomercury species passing through the entire length of the heating trap column and decomposing to Hg(0) (Liang, Bloom, and Bloom 1994). Under a helium flow rate of 45 mL·min^{-1} (rotometer set at 30), apply appropriate voltage to the coil around the column so that the column reaches 200° C from room temperature within 30 seconds. At BRL the heating is initiated by turning on the peak integrator. The organomercury species are desorbed and carried through the GC column that is held in an oven at 105° C. The species elute in order of increasing molecular weight, pass through the pyrolitic column, held at approximately 900° C at which point all organomercury species are converted into Hg(0), and detected by CVAFS. The sparging reaction vessels must be triple rinsed with DDW after each use to flush out reaction by-products which can interfere with subsequent purgings.

9. CALCULATIONS

BRL uses the following formulas for the calculation of monomethyl mercury in a given sample.

9.1. Mean Calibration Coefficient:

A calibration coefficient (CF) is calculated for each standard used in the calibration as follows.

$$CF = CS_{pgMMHg} / (CS_{PA} - EB_{PA})$$

where CS_{pgMMHg} is the calibration standard measured in picograms of methyl mercury, CS_{PA} is the peak area or peak height obtained during the analysis of the standard, and EB_{PA} is the mean peak area obtained during the analyses of all of the ethylation blanks. The mean calibration coefficient (CF_{avg}) is then calculated for all of the standards used in the calibration.

9.2. Measured methyl mercury in the sample preparation:

The amount of methyl mercury present in the analyzed volume of the sample preparation is calculated using the equation:

$$MMHg_{measured\ pg} = (A_{PA} - EB_{PA}) \cdot CF_{avg}$$

where A_{PA} is the peak area obtained during the analysis of the sample preparation.

9.3. Total methyl mercury in the sample preparation:

The total amount of methyl mercury present in the sample preparation is calculated using the equation:

$$MMHg_{total\ pg} = (MMHg_{measured\ pg}) \cdot V_A / V_D$$

where V_D is the final dilution volume of the sample preparation in mL and V_A is the volume analyzed of the sample preparation in mL.

9.4. Concentration of methyl mercury in the sample:

The final concentration of methyl mercury in the sample is calculated using the equation:

$$MMHg_{conc} = (MMHg_{total\ pg} - MB_{total\ pg}) / V_o$$

where $MB_{total\ pg}$ is the average total picograms of methyl mercury present in the method blanks and V_o is either the volume of the prepared sample measured in mL (aqueous samples) or the weight of the prepared sample measured in mg (solid samples). Therefore, the final concentration of methyl mercury in the sample is reported in units of ng/L for aqueous samples and in units of ng/g for solid samples.

NOTE: The total picograms of mercury present in each method blank is calculated using the same formula used to calculate the total picograms of methyl mercury in the sample preparation.

9.5. Empirically derived correction factor

Brooks Rand does not routinely recovery correct sample results to account for the fact that the distillation procedure is not 100% efficient in recovering methyl mercury. If a client requests that the results for any distilled samples are recovery corrected, then those results are multiplied by an empirically derived correction factor that is based on the average recovery of the appropriate QCS or CRM sample. If an appropriate QCS or CRM sample is not available, the correction factor is based on the average recovery of the spikes made to samples with a similar matrix to the sample of concern.

The correction factor is calculated using the following equation:

$$F = 100 / R$$

where F is the empirically derived correction factor and R is the running mean of the recoveries of the last 30 quality control samples or matrix spikes.

Brooks Rand does not use the IPR and OPR samples to calculate the correction factor since, unlike the client samples and quality control samples, these samples are not distilled.

10. QUALITY CONTROL

10.1. Refer to EPA Method 1630, Section 9.0 for a detailed description of the quality control procedures employed at BRL for this method.

10.2. All quality control data should be maintained and available for easy reference and/or inspection.

10.3. Each analyst must perform an initial demonstation of capability (IDOC) for the analysis of methyl mercury prior to the analysis of any client samples. The IDOC consists of a method detection limit (MDL) study for each preparation method and/or matrix following the procedure in 40 CFR 136, Appendix B and an initial precision and recovery (IPR) study following the procedure in EPA Draft Method 1630, Section 9.2.2. The acceptance criteria and run sequence for the IDOC can be found in Table 3 in Section 12 of this SOP.

10.4. Calibration data must be composed of a minimum of 1 ethylation blank (BRL analyzes 4 ethylation blanks prior to analyzing the calibration standards) and a minimum of 5, preferably 6, standards (See Section 8.2 of this SOP). Such a calibration should be run when stock standards have been remade, conditions have changed, or initial calibration check (ICV) or ongoing precision and recovery (OPR) as defined in Section 10.5 do not yield acceptable recoveries.

10.5. Ongoing precsion and recovery (OPR) solution prepared at a concentration of 0.5 ng/L and followed by an ethylation blank must be analyzed prior to and at the end of the analysis of each analytical batch. Additionally, BRL analyzes an independent calibration check (ICV) solution obtained from a source independent from that used to obtain the calibration standard and prepared at a concentration of 0.5 ng/L prior to the analysis of each analytical batch. The criteria for the recovery of the OPR and the ICV solutions is 67-133%. All ethylation blanks must contain no more than 2.0 pg MMHg.

10.6. Matrix spike/matrix spike duplicate (MS/MSD) analysis should be performed once per every 10 client samples or once per batch, whichever is greater. A matrix spike sample is defined as an aliquot of homogenized sample that has a known amount of analyte added to it. The matrix spike sample is then processed through the entire preparation and analytical procedure. Bias is then determined by calculating the percent recovery of the known amount using the following formula:

$$\text{Percent Recovery} = 100 * (\text{spiked sample result (conc.)} - \text{sample result (conc.)}) / (\text{amount spiked})$$

The criterion for spike recovery is determined by control charts and is different for each matrix type. The specific matrix spike recovery criteria for each matrix type can be found in Table 4 in Section 12 of this SOP.

The relative percent difference between the MS and the MSD is calculated using the following formula:

$$RPD = 200 \cdot (|MS\text{-}MSD|) / (MS + MSD)$$

The RPD for the MS/MSD pair must meet the criterion for each of the matrix types found in Table 4 in Section 12 of this SOP.

10.7. Method duplicates are prepared and analyzed upon client request. The RPD between duplicate samples is calculated using the same formula as used to calculate the RPD between the MS and MSD samples. The acceptance criterion for aqueous samples is RPD $\leq$ 35% or ± the PQL if the native result is < 5 times the PQL and the acceptance criterion for solid samples is RPD$\leq$ 35% or ± 2 times the PQL if the native result is < 5 times the PQL. If the acceptance criterion for duplicate analysis is not met for either samples or matrix spike samples, then the system performance is unacceptable. The problem must be corrected and the batch must be reanalyzed.

10.8. Field duplicates are analyzed at the client's discretion. The acceptance criterion for field duplicate analysis is the same as that used for method duplicate analysis. The client must be notified immediately anytime that the acceptance criterion for field duplicates is not met.

10.9. A minimum of 3 method blanks (MB) per batch must be analyzed. Method blanks are prepared identically to the preparation of samples, including the addition of any preservation chemicals that may have been added to the samples. Therefore, a MB prepared for a batch of fresh water samples would be "preserved" with 0.4% (v/v) 11.6 M HCl. The acceptance criteria for the method blanks is an average less than the current MDL (See Table 1 of Section 12) and a standard deviation less than 1/3rd the current MDL or the highest method blank result must be less than 1/10th the lowest sample result.

10.10. Quality control samples (QCS) are prepared and analyzed with each batch at a frequency of once per every 10 client samples or once per batch, whichever is greater. Aqueous QCS are prepared by spiking a method blank sample with a standard obtained from a source other than the source of the standard used in the calibration. The QCS is then distilling as per an aqueous sample. The acceptance criterion for the recovery of the QCS is identical to the acceptance criterion for the recovery of a matrix spike to an aqueous sample as shown in Table 4 in Section 12.

NRC or NBS certified reference materials for methyl mercury in tissue and sediment samples must be analyzed at a frequency of once per every 10 client samples or once per batch, whichever is greater. Criteria for CRM recoveries are determined by control charts. If control charts are not available then CRM results should be within 35% of the certified value for the analysis to be considered valid. CRM accuracy results not meeting this criterion shall be reprepared and reanalyzed or qualified at the discretion of the lab director. Currently, there are not any water based CRMs available.

11. REFERENCES

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Bloom, N.S.; Colman, J.A.; and Barber, Lee. (1997) "Artifact Formation of Methyl Mercury during Aqueous Distillation and Alternative Techniques for the Extraction of Methyl Mercury from Environmental Samples." *Journal of Analytical Chemistry* 358:371-377.

Bloom, N.S. (1989) "Determination of Picogram Levels of Methylmercury by Aqueous Phase Ethylation, Followed by Cryogenic Gas Chromatography with Cold Vapor Atomic Fluorescence Detection." *Canadian Journal of Fisheries and Aquatic Sciences.*

Long, S.J.; Scott, D.R.; and Thompson, R.J. (1973) "Atomic Absorption Determination of Elemental Mercury Collected from Ambient Air on Silver Wool." *Anal Chem.* 45: 2227-2233.

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EPA Draft Method 1630 (January 2001) "Methyl Mercury in Water by Distillation, Aqueous Ethylation, Purge and Trap, and CVAFS."

EPA Method 1669 (April 1995) "Sampling Ambient Water for Trace Metals At EPA Water Quality Criteria Levels."

12. TABLES and BENCHSHEETS

Table 1 Current Method Detection Limits and Minimum Levels Determined at BRL for the Analysis of Methyl Mercury Using EPA Method 1630

Matrix	Preparation Method	Method Detection Limit (MDL)[1]	Minimum Level (ML)
Water	Distillation	0.075 ng/L	0.25 ng/L
Sediment Sludge	Distillation	0.02 ng/g	0.05 ng/g
Biota	Digestion	1.8 ng/g	4.2 ng/L

NOTES
1. MDL as determined by the procedure 40 CFR Part 136. Appendix B.

Table 2 Summary of Control Chart Data Collected from January 2001 to February 2003 for the Analysis of Methyl Mercury Using EPA Method 1630

QA Sample	Matrix	Mean[1] Recovery (%)	Warning Limit (%) Mean ± 2 StDev	Control Limit (%) Mean ± 3 StDev
ICV	ALL	98.7	77.5 – 120.0	66.8 – 130.6
OPR	ALL	100.6	82.1 – 119.1	72.9 – 128.3
Matrix Spikes	Water	76.0	60.6 – 91.5	52.8 – 99.2
Matrix Spikes	Sed/Sludge	83.1	51.3 – 114.7	35.4 – 130.8
Matrix Spike	Biota	104.9	82.8 – 127.0	71.7 – 138.1
LFB	Water	78.7	70.6 – 86.7	66.6 – 90.8
CRM[2]	Sed/Sludge	90.1	69.1 – 111.2	58.5 – 121.8
CRM[3]	Biota	99.6	76.2 – 123.1	64.5 – 134.8
QA Sample	**Matrix**	**Mean RPD**	**Warning Limit (%) Mean ± 2 StDev**	**Control Limit (%) Mean ± 3 StDev**
Duplicates[4]	Water	11.7	29.1	37.8
Duplicates[4]	Sed/Sludge	12.4	25.9	32.7
Duplicates[4]	Biota	11.1	30.2	39.8

NOTES
1. Average recoveries for distillations (water and sediment samples) are less than 100% since results have not been recovery corrected using an empirically derived correction factor.
2. BCR-580 (Marine Sediment) is the CRM used for sediments.
3. DORM-2 (Dogfish Muscle) is the CRM used for most biota samples.
4. Duplicates criteria is for both duplicates of the native sample and duplicates of the matrix spike.

Table 3 Quality Control Acceptance Criteria and General Analytical Run Sequence for the Initial Demonstration of Capability for the Analysis of Methyl Mercury

Run	Run Name	Section Name	Analyze	Requirements
1	Ethylation Blank	Calibration	Ethylation Blank	each ≤ 2 pg
2	Ethylation Blank		Ethylation Blank	
3	Ethylation Blank		Ethylation Blank	
4	Ethylation Blank		Ethylation Blank	
5	10 pg std	Calibration	0.1 ng/L	RSD of Avg. CF ≤ 15% Recovery of Low Standard 65-135%
6	50 pg std		0.5 ng/L	
7	100 pg std		1.0 ng/L	
8	250 pg std		2.5 ng/L	
9	1000 pg std		10.0 ng/L	
10	2000 pg std		20.0 ng/L	
11	Method Blank	Contamination Check	Method Blank	Mean < Target MDL
12	Method Blank		Method Blank	
13	Method Blank		Method Blank	
14	IPR std (25pg)	Initial Precision and Recovery	0.5 ng/L	Ave. recovery 69-131%, RSD ≤ 31%
15	IPR std (25pg)		0.5 ng/L	
16	IPR std (25pg)		0.5 ng/L	
17	IPR std (25pg)		0.2 ng/L	
18	MDL sample	Method Detection Limit	Appropriate matrix spiked at a level of 1 – 5 times the expected MDL	Calculated MDL no greater than 5 times the spike level and RSD > 10%
19	MDL sample			
20	MDL sample			
21	MDL sample			
22	MDL sample			
23	MDL sample			
24	MDL sample			
25	OPR std (25pg)	Ongoing Precision and Recovery	0.5 ng/L	Recovery 67-133%
26	Ethylation Blank	Contamination Check	Ethylation Blank	≤ 2 pg

NOTES:
1. All standards and samples are corrected for mean ethylation blank.
2. All samples are corrected for mean method blank blank.

Table 4 Quality Control Acceptance Criteria and General Analytical Run Sequence for the Analysis of Methyl Mercury

RUN	Analyze	Description	Requirements
1	Ethylation Blank (EB)	Contamination Check	≤ 2.0 pg
2	Ethylation Blank (EB)		
3	Ethylation Blank (EB)		
4	Ethylation Blank (EB)		
5	10 pg std	Calibration Curve	RSD of Avg. CF ≤ 15% Recovery of Low Standard 65-135%
6	50 pg std		
7	100 pg std		
8	250 pg std		
9	1000 pg std		
10	2000 pg std		
11	ICV (independent calibration verification) (500 pg)	Precision and Recovery	70 – 130% recovery
12	OPR std (25 pg)	Ongoing Precision and Recovery	67 – 133% recovery
13	Ethylation Blank	Contamination Check	≤ 2.0 pg
14	Method Blank 1 (MB-1)	Contamination Check	Mean ≤ MDL StDev ≤ MDL
15	Method Blank 2 (MB-2)		
16	Method Blank 3 (MB-3)		
17	Sample 01[2]	Native Sample	
18	Sample 01MS	Matrix Spike	Rec = 60 – 120%[3] for aqueous and sediment samples and Rec = 75 – 135% for biota samples
19	Sample 01MSD[4]	Matrix Spike Duplicate	Rec = 60 – 120%[3] for aqueous and sediment samples and Rec = 75 – 135% for biota samples; RPD ≤ 35%
20	Sample 02	Native Sample	
21	Sample 02MS	Matrix Spike	Rec = 60 – 120%[3] for aqueous and sediment samples and Rec = 75 – 135% for biota samples
22	Sample 02MSD[4]	Matrix Spike Duplicate	Rec = 60 – 120%[3] for aqueous and sediment samples and Rec = 75 – 135% for biota samples; RPD ≤ 35%
23	Sample 03	Client Sample	
24	Sample 04	Client Sample	
25	Sample 05	Client Sample	
26	Sample 06	Client Sample	
27	QCS or CRM	Precision and Recovery	Rec = 60 – 120%[3] for aqueous and sediment samples and Rec = 65 – 135% for biota samples
28	Sample 07	Client Sample	
29	Sample 08	Client Sample	
30	Sample 09	Client Sample	
31	Sample 10	Client Sample	
32	Sample 11	Native Sample	
33	Sample 12	Client Sample	
34	OPR std (25 pg)	Ongoing Precision and Recovery	67 – 133% recovery
35	Ethylation Blank	Contamination Check	≤ 2.0 pg

NOTES

1. The calibration curve may be adjusted depending on the expected range of samples (i.e. seds and biota 10pg-5000pg)
2. Any known field or equipment blanks should be analyzed prior to other samples. The acceptance criterion for these samples is a result ≤ the ML.
3. Not recovery corrected
4. Clients may request method duplicate analysis, in which case lab personnel will be notified.

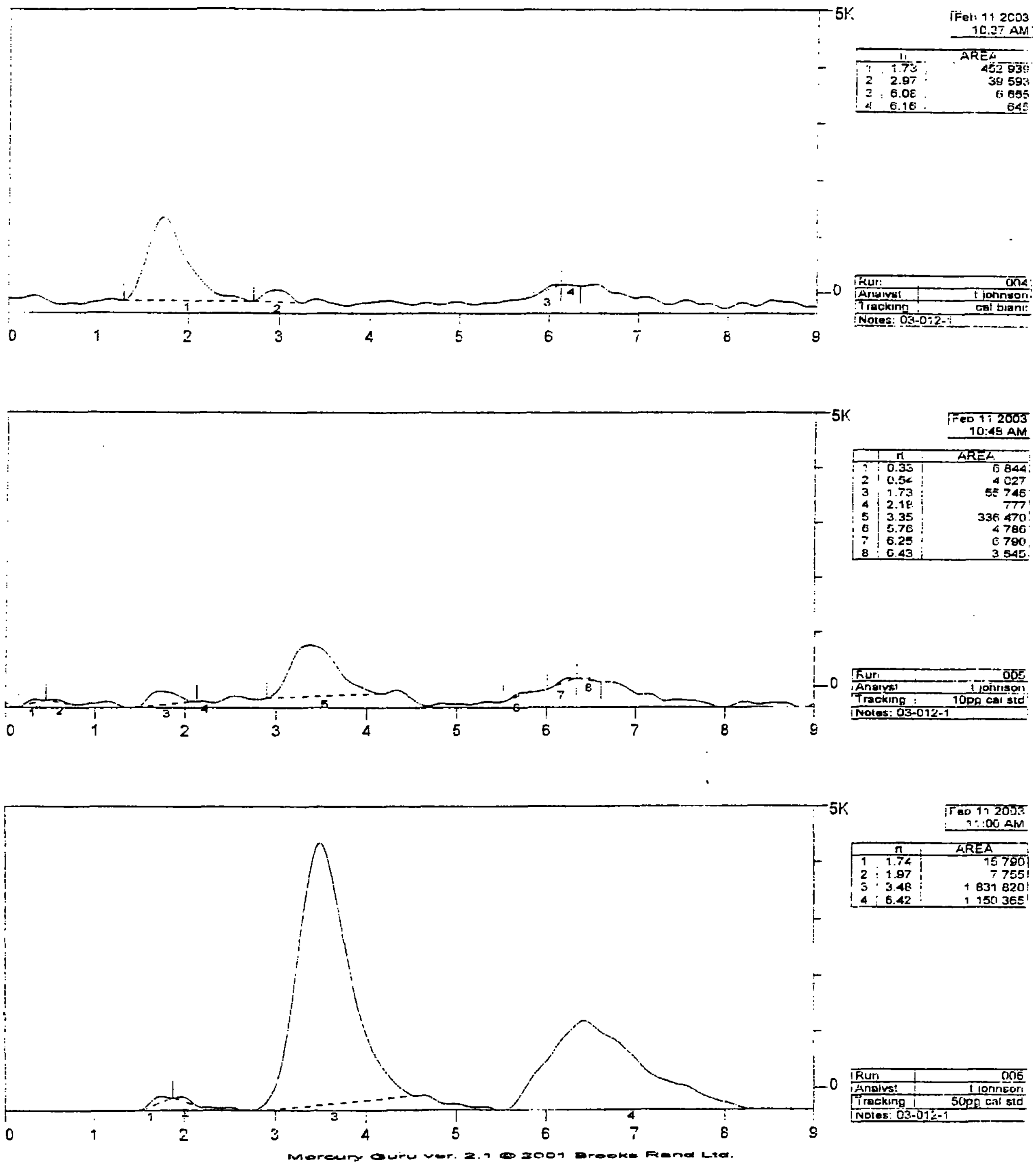

Figure 3 Example of Typical Chromatograms Generated by the Mercury Guru Software During the Analysis of Methyl Mercury Using EPA Method 1630

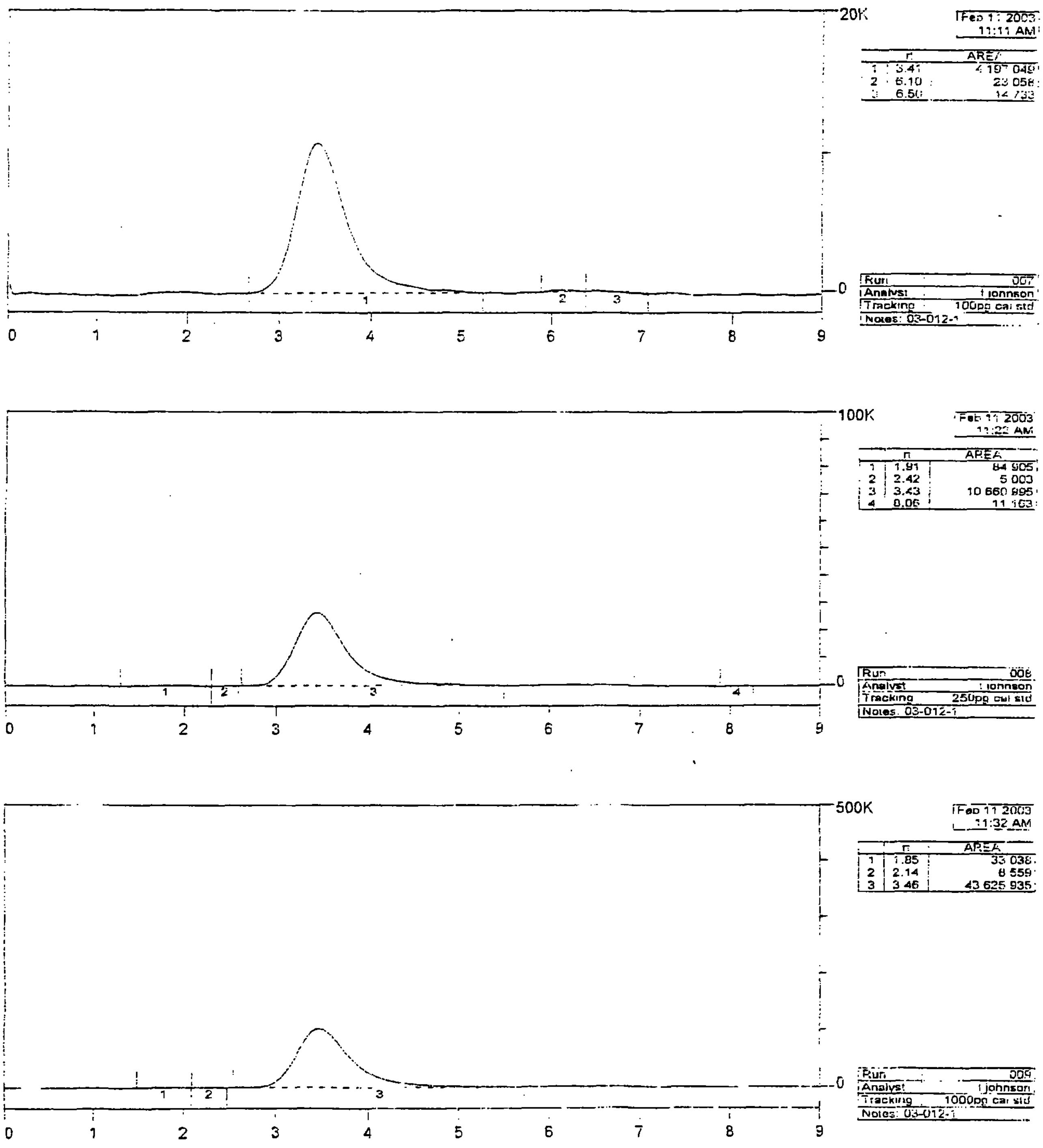

Figure 4 Additional examples of Typical Chromatograms Generated by the Mercury Guru Software During the Analysis of Methyl Mercury Using EPA Method 1630

MM-Hg Method BR-0011 (CVAFS)
Revision #: 008

Batch: ____________________ Matrix: ______________________________

Tracking #(s): ____________________ Preparation Date: ______________________________

Project #(s): ____________________ Prepared By: ______________________________

QA: ____________________ Page _______ of _______

Sample I.D.	Aliquot (mL) / (mg)	Sample I.D.	Aliquot (mL) / (mg)

Matrix Spike/Matrix Spike Duplicate

Sample I.D.	MMHg Standard I.D.	MMHg Standard Conc. (ng/mL)	MMHg Standard Vol. used (mL)	Matrix Spike Conc. (ng/L) / (ng/g)

Quality Control Sample (QCS)

QCS I.D.	LFB/CRM I.D.	LFB/CRM Conc. (ng/L) / (ng/g)	LFB/CRM Vol./Mass (mL)/(mg)

KOH in Methanol Reagent I.D. # (Biota): ____________________

Final Dilution Volume: ______________________________

Comments: ____

____

____

MMHg Analysis Sheet

☐MMHg ☐Other:_____ Batch:__________________ Matrix:____________

Analyst:________________ Calibration Blank $\bar{X}$:__________ Blank Corr. Calib. Coef. $\bar{X}$:__________
Date:________________ Method Blank $\bar{X}$:__________ Pretrap:__________ RSD:__________
Instrument ID = __________ Standards : ng/mL __________ Buffer:__________ N=:__________
QA: Full☐ Standard☐ : ng/mL __________ NaBEt:__________ r:__________

Run	Trap	Sample ID	Sample vol/wt	Dilution Volume	Analyzed volume	Raw PA	pg	Result

Calculation Equation:______________________________
Comments:______________________________

Independent Source Std.:__________________ Method SRM:__________________

MMHg Analysis Sheet

Page___of___

Batch:____________ Analyst:____________ Date:___________

Run	Trap	Sample ID	Sample vol/wt.	Dilution Volume	Analyzed volume	Raw PA	pg	Result ____

Comments:____

COPY

SOP #BR-0300

Receipt of Samples

Brooks Rand, Ltd.

Written 3/9/93
Revised 7/17/01

Revision 005

Reviewed ______________________

____

____

____

R. Mangon
Operations Manager

12/21/01
Date

F. McFarland
QA Manager

12/12/01
Date

Senior Scientist

12/12/01
Date

Tiffany Kao
Scientist (if applicable)

7/17/01
Date

Receipt of Samples

1. DESCRIPTION
   A. Definition: Documenting the arrival and status of samples.

   B. Scope: To ensure all samples and necessary information is present, and any discrepancies are noticed and quickly resolved.

   C. Summary: When samples arrive the samples custodian or the designated alternate checks and documents sample conditions, assigns BRL sample identification numbers, logs all sample information into the BRL "Tracking" database, preserves, and stores all samples.

2. EQUIPMENT
   A. Electronic Sample Receiving System: Consisting of PC computer with MS Access® and BRL "Tracking" Database.

   B. Sample Receiving Log Book: Three ring binder for storage of up to 100 original, computer generated sample receiving log sheets.

   C. Sample Storage Areas: Refrigerator or freezer for solid samples and refrigerator or non-metal cabinet for water samples.

   D. Preservation Reagents: HCl, BrCl, H_2SO_4, HNO_3 etc. for preserving water samples.

3. PROCEDURE
   A. Sample Analysis Request: Clients are requested to notify the Project Manager or Sample Custodian prior to sample shipment. Any time the sample custodian is not available the designated Alternate Sample Custodian must perform the work. Both the Sample Custodian and Alternate Sample Custodian are referred to as Sample Custodian for the remainder of this SOP. Clients are requested to fax the Chain of Custody (C.O.C.) or other sample identification documentation to BRL at the time they send samples. This allows the Sample Custodian to be prepared for the receipt of samples the following day. The client may send the C.O.C. form along with the sample shipment instead of faxing the form if they so choose.

   B. Sample Receiving: Samples are delivered to the sample receiving door. The Sample Custodian then documents all information on the sample receiving log form (see Exhibit A) in the BRL "Tracking" database. To obtain this information, the sample custodian checklist (see Exhibit B) should be followed and filled out during the process of receiving.

   Before the sample shipping container is opened, its condition should be documented as either being intact or any damage should be described. The custody seal (if present) information should also be recorded before opening the container. The airbill should be removed from the cooler and kept for documentation. The shipping container is then opened. All samples, regardless of whether they are known to be non-hazardous or not, are unpacked in the fume hood located in the northeast corner of the downstairs laboratory. It is up to the Project Manager or designee to let the Sample Custodian know if hazardous samples are to arrive, and the specific hazards associated with them.

Sample bottle conditions include both temperature conditions and physical conditions (either intact or a description of the problem(s) with samples). It is very important that the Sample Custodian is aware of the preferred condition for the samples. Solid samples should be received dried, cold or frozen. Water samples should be shipped on ice and received cold. Generally, the preservation temperature of solid and water samples should be less than 4°C. The temperature of the cooler should be taken and recorded by placing a thermometer inside the cooler or in a temperature blank (provided by the client), closing the lid, and allowing the temperature to equilibrate. After this time, the cooler should be reopened and the temperature recorded.

Teflon sample bottles (for water) are engraved with a unique bottle ID number, which can be used for sample identification. When a bottle is removed, the number engraved on the bottle should be matched with the number written on the bag. All information on sample bottles (or tags) should be checked against the C.O.C. or other documentation provided by the client. If this information does not match or any other significant problems are observed (e.g. sample bottle not intact), a non-conformance form (see SOP #BR-1204) should be filled out and the Project Manager should be notified. It is then the Project Manager's duty to contact the client to resolve discrepancies. Minor discrepancies (e.g. sample received outside of preservation temperature or time) can be noted on the non-conformance form and resolved by the Project Manager at a later time.

C. <u>EPA Sample Log-In Sheet (Exhibit C EPA form DC-1)</u>: This form must be filled out during receipt of EPA samples when required by the client. The Project Manager will notify the Sample Custodian when this form is required. Each section is completed by either filling in the appropriate information where asked, or circling the appropriate choice. If any of the items/descriptions marked with an * are circled the Sample Management Office (SMO) must be contacted and the discrepancy resolved. A record of resolution then must accompany this sample log-in sheet.

D. <u>Preservation</u>: If any filtration or volatile mercury analysis of water samples is required, this should be performed before preservation of samples (see SOP BR-0104 for filtration and Draft SOP BR-0005 for volatile mercury analysis). All samples should be preserved in accordance with the preservation instructions in each appropriate analytical methodology or in accordance with the client's requirements (see Exhibit D for an outline of preservation instructions). Typically, water samples that arrive for only total Hg, EPA method 1631, analysis are preserved with 0.5% BrCl. Water samples for only MMHg analysis are preserved with 0.8% HCl. Samples for Se and/or Se speciation are typically preserved with 0.8% HCl to pH < 2. Samples for As and/or As speciation are also preserved with 0.8% HCl and typically stored at 4° C. Solid samples do not need acid preservation but are preserved by being stored at <-10°C. The Project or QA Manager should be consulted if there is any question regarding sample preservation. For all water samples requiring acidification for preservation, the pH should be checked and documented as being less than pH 2. All preservation components should be documented including the lot number of the reagent, the type of reagent, and amount used.

E. Entering the Sample into the Database: For each current project, the Project Manager maintains an active file of information specific to the project in the BRL "Tracking" MS Access database. When logging in samples, the Sample Custodian checks the contract information against the samples received to ensure that the work has been authorized and to ensure that there are no discrepancies between the work contractually approved by the client's accounting department and the work requested by the client's sampling team. If any discrepancies are found, the Project Manager should be immediately notified.

In the "Tracking" MS Access database, the samples are given a unique sample identification number as outlined by SOP #BR-0302. The sample ID number, bottle number, sample matrix, bottle size and analysis requested should be entered for each sample into the database. Once the information for the samples is completely entered in the database, the sample receiving log should be printed out along with the labels for the samples and an "Internal Custody for Original Samples" sheet. The Sample Custodian prints out a copy of the "Internal Custody for Original Samples" form found in the MS Access® Tracking database. This form is kept with the samples throughout their lifespan, from receipt to disposal. (See BR-0301, "Sample Custody and Maintenance")

F. Storage: After samples have been preserved, entered into the database, and labeled, they are stored in the appropriate sample storage locations along with the "Internal Custody for Original Samples" form. The Sample Storage Rooms (#1 and #2), the shop Freezer (#2), and the Sample Storage Cabinets (#1 through #8) each have a clear plastic folder to hold Internal Custody forms. Each time the samples are removed from their place of storage, it should be documented on the form, and the form is to remain with the samples at all times. See BR-0303, "Sample Storage and Disposal" for further information on sample storage.

Water samples requiring Hg analysis are usually stored in Storage Cabinet #1 through #7; most other analytes require refrigeration and are stored in BRL Refrigerator #2 or #3. Sediment and biota samples are generally preserved by freezing and are stored in BRL Freezer #4. All high level or hazardous samples must be clearly labeled as such and stored according to their hazard (i.e. flammable samples stored in a secured flammable storage cabinet, or high level mercury samples stored separately from low-level mercury samples--see SOP BR-0303).

G. Document Control: After samples are logged-in and stored, all sample information is place into a folder. The sample information includes the following: the C.O.C., a copy of the BRL Sample Receiving Log, the original airbill, a copy of the airbill and the non-conformance form (if applicable). The folder should be labeled with the tracking number, the project reference number, the date received, and the due date. The folder is then given to the Project Manager for review prior to being faxed to the client and filed in the "Active Customer" file located in the Project Manager's office. Information for each current project is kept in the "Active Customer" files and is sorted alphabetically by the project reference number. Also included in the "Active Customer" file is a form to track the number of bottles shipped to the customer and the number of bottles returned (see Exhibit E). The Sample Custodian needs to record the quantity and size of Teflon bottles sent to the client, as well as track the bottles as they are received.

The original BRL Sample Receiving Log sheets should be kept in a three ring binder at the sample receiving desk. After Sample Receiving Log sheets accumulate up to 100 tracking numbers, the Sample Custodian should bind these originals in the velo-binder and store them with the rest of the previously bound receiving sheets in the back laboratory.

BR-0300 -6
Revision 005
Exhibit A

Brooks Rand, LTD. Sample Receiving Log

Tracking #

Customer
Contact
Project Ref #

Collection Date
QA Level

Sample Condition
Shipping container intact?

Shipping container type
Shipping container temp
Shipping container coolant

Sample preservation
Acid lab #
Hg Concentration
Sample storage area

Sample Turnaround Time
Contract Turnaround Time days

Comments

Due Date:
Receiving Date:
Receiving Time:

Logged-in by:
Log-in Date:
Log-in Time:

Airbill present?
Airbill #
Carrier:

Custody seal present?
Custody seal intact?
COC Present?
COC Number:

Case #:
SDG #:

Analysis request form?

COC/Sample log agree?

Lab ID	Sample Tag #	Container #	Size	pH	Matrix/Sub-Matrix	Comments:
					#Error	

Analysis / Method

Sample Custodian signature Date

Sample Custodian Checklist

Type of Shipping Container:	Cooler		Cardboard Box		Other________
Condition of Shipping Container:	Intact		Damaged*		
Custody Seal Present:	Yes		No		
Custody Seal Intact:	Yes		No*		
Type of Coolant:	Blue Ice	Ice	Dry Ice		None
Temperature:	________°C**				
C.O.C. Present:	Yes		No		
C.O.C. Signed:	Yes		No	N/A	
ARF Present:	Yes		No		
Condition of Sample:	Intact		Damaged*		
Sample Tags Agree w/ COC/ARF:	Yes		No*		
Bottle/Container:					
Type:	Teflon	Poly	Glass	Other________	
Size:	___________mL				
Teflon Bottle #s:	______________________________				
Bottle Custody Seals Present:	Yes		No		
Airbill Present:	Yes		No	N/A	
Carrier:	UPS	FedEX	Airborne	Hand-delivery	Other________
Preservation:					
Chemical:	________________				
Concentration/Percentage:	________________				
ID #:	________________				
Final pH of Samples:	<2		Other________		
Storage Location:	Cab #1	Cab#2	Cab#3	Fridge#2	Freezer#4 Other_______
BRL Labels:	Completed				
Internal Custody for Original Samples:	Completed				
Bottle Accounting:	Recorded				
Faxing:	Completed				
Filing:	Completed				

* fill out non-conformance form
** if temperature >4°C for sediment/biota/water samples, fill out non-conformance form

Brooks Rand Log-in Sheet
for EPA samples

BR-0300 -8
Revision 005
Exhibit C

Received By (Print Name): ______________________ Log-in Date: ______________

Received By (Signature): ______________________

Case Number: ______________________

Sample Delivery Group No.: ______________________

SAS Number: ______________________

EPA SAMPLE #	CORRESPONDING SAMPLE TAG #	CORRESPONDING ASSIGNED LAB #	REMARKS CONDITION OF SAMPLE SHIPMENT, ETC.

REMARKS:

1. Custody Seal(s): Present/Absent* Intact/Broken
2. Custody Seal Nos.: ______________
3. Chain-of-Custody Records: Present/Absent*
4. Traffic Reports or Packing List: Present/Absent*
5. Airbill: Airbill/Sticker Present/Absent*
6. Airbill No.: ______________
7. Sample Tags: Present/Absent*

   Sample Tag Numbers: Listed/Not listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ______________
11. Time Received: ______________

Sample Transfer

Fraction:	______	______
Area #:	______	______
By:	______	______
On:	______	______

*Contact SMO and attach record of resolution

Reviewed By: ______________________

Date: ______________________

FORM DC-1

Logbook No.: ______________________

Logbook Page No.: ______________________

Analysis to be Performed	Preservative*	Holding Period (after preservation)	Container
H_2O			
Hg-Total, EPA 1631	0.5% BrCl	28 days	Teflon, Glass
Hg-Total, EPA 245.1	0.2% HNO_3	28 days	Teflon, Glass
MMHg-& Hg-Tg	0.8% HCl	28 days	Teflon, Glass
Se-Total, BR-0020 As-Total, BR-0020	0.8% HCl	28 days	Poly Bottle, Teflon, Glass
As(III), As(V), MMAs, DMAs BR-0021	0.8% HCl & 4°C	28 days	Poly Bottle, Teflon, Glass
Se(IV), Se(VI), BR-0023	0.8% HCl & 4°C	28 days	Poly Bottle, Teflon, Glass
All Metals, EPA 200.9	0.4% HNO_3	6 months	Poly Bottle, Teflon, Glass

*** ALL WATER SAMPLES CHECK pH < 2**

Analysis to be Performed	Preservative*	Holding Period (after preservation)	Container
Sediment			
As(III), As(V), MMAs, DMAs BR-0021	4°C	5days	Teflon, Glass
Hg-T, MMHg, As-T, As(inorg) Se-T	< -10°C	1 yr	Teflon, Glass
Biological			
Tissues	< -10°C	1 yr	Teflon, Glass

BR-0300 -10
Revision 005
Exhibit E

Brooks Rand, Ltd.

Teflon Bottle Accounting Form
Revision 002
3/29/01

Project # ____________

Teflon Size (mL)	Shipping		Receiving		Receiving		Receiving		Difference
	Date	Quantity	Date	Quantity	Date	Quantity	Date	Quantity	Quantity

COPY

SOP #BR-0301

Sample Custody Maintenance and Tracking

Brooks Rand, LLC

Written 3/15/93
Revised 6/10/02

Revision 004

Reviewed ______________________

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____

President	11/11/02 Date
QA Manager	10/18/02 Date
Senior Scientist	10/18/02 Date
Scientist (if applicable)	Date

Sample Custody Maintenance and Tracking

1. DESCRIPTION
   A. Definition: Maintaining and documenting possession and integrity of samples from receipt through disposal.

   B. Scope: Maintaining sample custody ensures sample integrity, data defensibility and documents tracking of samples through their entire life cycle.

   C. Summary: Sample custody is ensured by means of proper identification, tracking and security of samples, and all changes in custody are documented.

2. SAMPLE SECURITY
   A. Samples are identified as described in SOP BR-0302, *Sample Identification.* All samples are stored in accordance with the specific preservation and storage requirements outlined in the pertinent analytical SOPs (SOP #s BR-0001 through BR-0099) and in SOP BR-0303, *Sample Storage and Disposal.*

   B. All samples are labeled when received with the client project number, the date of receipt, the preservation, and the BRL sample identification number. Sample identification labels are kept on the sample containers at all times.

   C. Samples are kept in the custody of the responsible employee or in the custody of a secured sample storage area.

   Sample custody is defined as:
   the sample is in your possession, or
   the sample is in your view after it has been in your possession, or
   the sample was in your possession and you locked the sample in a secure area.

   D. The secure areas of Brooks Rand, LLC (defined as areas accessible to authorized personnel only) are as follows:
   Sample Storage Cabinets: located in the downstairs lab on the South facing wall. Cabinets are non-metallic (wood). Samples to be stored in this location include all original samples suitable for storage at room temperature.

   Sample Storage Room #1: located off of the hallway leading to the downstairs bathroom (Southmost corner of the building). This sample storage room contains BRL Refrigerator #2 and Freezer #4, and will be used for all samples (original and preparations) requiring cold storage of either 4°C or <-10°C.

   Sample Storage Room #2: located adjacent to Sample Storage Room #1. This sample storage room will be used to store all sample preparations and performance evaluation (PE) samples suitable for storage at room temperature.

Freezers #2: These freezers are located in the NW corner of the downstairs and will be used for long term storage (archival) of original solid samples after analysis is complete. BRL policy is to archive client solid samples for a minimum of one year prior to disposal.

All visitors are required to sign-in at the front door and must be accompanied by a laboratory employee while in the laboratory. A copy of the visitor sign-in log sheet is included as exhibit A. The laboratory is kept locked outside of normal business hours.

In addition to the above mentioned sample custody maintenance steps, each stage of the process for a batch of samples is documented on the Sample Processing Form (see SOP # BR-0304) and is logged in on computer by the responsible employee. This serves as a further tracking device to monitor sample status.

3. PROCEDURE FOR TRACKING CUSTODY OF ORIGINAL SAMPLES

When samples are received, a "Internal Sample Custody for Original Samples" form shall be filled out (see Exhibit B). Original samples shall be tracked by the tracking number assigned to them upon receipt. This tracking number and the client project # is documented on the custody form. In addition, all activity (receipt), location (sample receiving), initials, date, and time shall be recorded in respects to the sample IDs. Once samples have been logged in, the samples are transferred to the appropriate storage location. The custody form is then used to document Activity (storage), location (i.e. sample storage cabinet), initials, and date and time of custody transfer. The custody form is then placed in the holder located on the outside of the sample storage location.

Whenever samples are removed from the storage location for sample processing (i.e. sample preparation), the custody form is then used to document this custody transfer. Similarly, when this activity is completed and samples are transferred back to storage, this is also documented. When samples are disposed of this information is documented on the custody form as the last entry.

In summary, every transfer of custody should be documented. Custody forms must always accompany the set of samples either physically with the samples when they are in the possession of an analyst or in the holder when they are in storage.

4. PROCEDURE FOR TRACKING CUSTODY OF SAMPLE PREPARATIONS

When samples are being prepared, a "Internal Sample Custody for Sample Preparations" form (See exhibit C) must be generated. Documentation on this form shall include the Batch number, analyte and analytical method number, sample ID numbers, activity, location, initials, date and time. Each time custody is transferred (either to another employee or to storage), the activity, location, initials, date and time are documented.

5. QUALITY ASSURANCE

Each person is responsible for filling out the appropriate information for the activity that they performed. Upon disposal of a set of samples, the custody forms of the original samples and the sample preparations are then considered complete and filed away for document archival in the Document Storeroom.

Exhibit A

Brooks Rand, LLC
Visitor Sign-in Sheet

Date	Visitor Name	Company	Employee Contact	Time In	Time Out

INTERNAL CUSTODY FOR ORIGINAL SAMPLES

Tracking #: Project Reference #:

Activity									
Location									
Initials									
Date									
Time									
Sample ID									

INTERNAL CUSTODY FOR SAMPLE PREPARATIONS

Batch #: Analysis: Method #:

Activity									
Location									
Initials									
Date									
Time									
Sample ID									

COPY

SOP #BR-0303

Sample Storage and Disposal

Brooks Rand, LLC

Written 3/15/93
Revised 10/10/02

Revision 004

Reviewed ______________________

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______________________ 11/11/02
President Date

______________________ 10/21/02
QA Manager Date

______________________ 10/21/02
Senior Scientist Date

______________________ ______________
Scientist (if applicable) Date

Sample Storage and Disposal

1. DESCRIPTION
   A. Definition: The location and duration of storing samples, documentation and disposal.

   B. Scope: To ensure the integrity of the samples, and to ensure proper disposal of samples and sample waste.

   C. Summary: Water samples are stored in the designated non-metal cabinet or refrigerator. Solid samples are stored in a calibrated freezer for at least one-year unless otherwise specified by the client. Samples are disposed, once approval is obtained, and disposal is documented.

2. EQUIPMENT
   A. Storage facilities and Equipment: Calibrated freezers, calibrated refrigerator, non-metal storage cabinet

   B. Disposal Facilities and Equipment: Disposal containers (30 gallon drums and 5 gallon buckets) and spill containment equipment

3. STORAGE
   A. Receipt: Samples are received in the downstairs laboratory (see SOP BR-0300, Receipt of Samples). Prior to storage, an "Internal Custody for Original Samples" form is created for each shipment, which will document each step of a sample's life cycle--from receipt to disposal. Similarly, an "Internal Custody for Sample Preparations" will be created for each group of samples as they are batched for analysis preparation, documenting the life cycle for each batch of preparations. (See BR-0301, "Sample Custody Maintenance.")

   B. Water samples: Water samples are preserved when received (see SOP BR-0300). After receipt, samples are stored in the designated storage cabinets or refrigerator (unless otherwise requested by the client or lab manager) until they are transferred to other personnel for preparation or analysis. On occasion, it may be necessary to store samples in Sample Storage Room #2 due to the limited storage capacity of the sample storage cabinets. The designated storage cabinets at Brooks Rand are located in the downstairs front laboratory in the southeast corner above the counter, and are marked and identified as "Sample Storage Cabinet #1" and "Sample Storage Cabinet #2". These cabinets should only be opened when necessary to minimize UV exposure and to restrict airflow and particulates. All water samples are stored for a minimum of one month from the time of receipt at Brooks Rand, unless requested otherwise.

   C. Solid samples: After receipt, solid samples are stored in the freezer located in Sample Storage Room #1 (BRL Freezer #4). After samples have been analyzed and the data is reviewed and deemed acceptable, the remaining portions are stored in one of the freezers located in the downstairs northwest corner of the building for long term storage (Freezer #2). Solid samples are stored for a minimum of one year from the time of receipt unless requested otherwise.

D. Air samples: Air samples collected on traps should be stored in a ziplock bag with the Teflon caps on the ends. The bagged traps can be stored at room temperature in Sample Storage Room #2 until analysis. Organic mercury samples on carbon traps should be analyzed within three days from collection. Total and elemental mercury samples collected on gold-coated silica traps can be stored for up to three months before analysis. For air samples collected on either carbon or gold coated silica traps, once the trap has been analyzed there is no sample remaining and therefore there is no storage for samples after analysis.

E. Volatile analysis: Samples for analysis of volatile mercury species are kept at 4°C in BRL Refrigerator #2 until analyzed. These analyses should be performed within 48 hours of sample collection. Once the analyses are complete, these samples should be treated appropriately depending on remaining analyses. If any further analyses are to be performed, the samples should then be preserved and stored appropriately.

F. Sample preparations: After preparation, each sample batch will be stored in Sample Storage Room #2 until analysis. After the batch has been analyzed, the sample batch will be returned to Sample Storage Room #2 until the lab manager releases them for disposal.

G. High level and hazardous samples: Brooks Rand must be notified if samples are suspected to be high in mercury or other analytes. All samples determined (or suspected) to be extremely high level, must be stored separately in a secure storage area outside of the analytical laboratory to ensure cross-contamination of the clean lab or other samples does not occur. Sample Storage Room #2 may be used for high level samples provided that there are no low-level water samples or preparations being stored in this storage room at the same time. BRL Freezers #2 or BRL Refrigerator #2 may also be utilized for high-level sample storage (depending on the sample matrix). If a client notifies BRL that samples may be hazardous in properties that would not contribute to clean lab contamination, but could possibly compromise worker safety, those samples must be handled as hazardous. All high level or hazardous samples must be clearly labeled as such and stored according to their hazard (i.e. flammable samples stored in a secured flammable storage cabinet, or high level mercury samples stored separately from low-level mercury samples). All laboratory workers receive yearly Right-To-Know training; in addition, at least one laboratory worker should be trained in the handling of hazardous waste, and will provide yearly training to other workers in the proper safe handling procedures for hazardous substances (see the BRL Chemical Hygiene Plan for further details).

4. SECURITY

The secure areas of Brooks Rand, LLC (defined as areas accessible to authorized personnel only) are the analytical laboratories, the designated sample storage cabinets, the sample storage freezers, and the sample storage rooms. All visitors are required to sign-in and must be accompanied by a laboratory employee while in the laboratory. The laboratories and office spaces are kept locked outside of normal business hours. The sample storage rooms and cabinets are unlocked only to retrive samples or sample preparations, but are otherwise kept locked at all hours.

5. HANDLING OF SAMPLE PREPARATIONS

A. After a batch is analyzed, the preparations for a batch shall be stored in Sample Storage Room #2, clearly labeled with the batch number, and stored in such a way that the batch is kept intact and separate from samples in other batches. This will enable the Sample Custodian to easily locate the specific batches that have been designated for disposal.

B. Occasionally, Brooks Rand LLC will receive samples that are known to contain dangerous properties other than the metals listed above. One example is a sample known to contain background levels of radioactivity (See SOP #'s BR-1600 and BR-1601 for Low Level Radioactive Waste storage and packaging for disposal). The sample preparations for hazardous samples should be clearly marked at the time of preparation for the safety of lab employees and so that the sample custodian may dispose of the preparations in a manner consistent with the disposal of the original samples.

6. DISPOSAL (or TRANSFER)

A. Sample Preparations: Once samples have been analyzed, reported, and either the data has been reviewed to be acceptable or has been determined that there is no value in reanalyzing the sample preparations, the sample preparations may be disposed. Sample preps may also be transferred for long time storage, upon client's request. The Lab Manager (or designee) will notify the Sample Custodian of any batches ready for disposal or transfer. The Sample Custodian shall retrieve the appropriate batch(es) and dispose or transfer the sample preparations accordingly (see Section 6C). The disposal of each batch shall be documented on the "Internal Custody for Sample Preparations" form (see section 7).

B. Original Samples: After completion of a report for a particular set of samples (one or more tracking numbers), the original samples may be either transferred to long-term storage or disposed of, if approved by the Lab Manager or the client. The Lab Manager will notify the Sample Custodian which original samples to dispose of or transfer. The Sample Custodian shall dispose (or transfer) the samples accordingly (see Section 6C). When samples are disposed, the "Internal Custody for Original Samples" log sheet should be signed off appropriately (see section 7).

C. Disposal Guidelines: Current limits for the analytes most frequently tested at BRL are as follows:

Table 1. - Disposal Limits

Analyte	Matrix	Limit (grab)	Source of Information*
As	Water	4.0 mg/L or ppm	King Co. Water Pollution Control Div.
As	Solids	5 µg/g or ppm	King Co. Solid Waste Div.
Hg	Water	0.2 mg/L or ppm	King Co. Water Pollution Control Div.
Hg	Solids	0.2 µg/g or ppm	King Co. Solid Waste Div.
Se	Water	1 mg/L or ppm	King Co. Water Pollution Control Div.
Se	Solids	1 µg/g or ppm	King Co. Solid Waste Div.

*Information updated 5/97. King County Water Pollution Control Division was formerly part of Metro.

1. Routine Disposal – Samples below the disposal limits.
   a) Water Samples and Acid Digestion - Water samples (including preparations) and acid digested solid samples that are not hazardous may be disposed of down the drain. All acidic samples must be neutralized with Soda-Ash prior to disposal. Original sample and sample preps that contained BrCl must be further neutralized with 30% hydroxalmine hydrochloric (NH_2OH-HCl). All labels identifying the client must be removed from the bottles. All neutralized samples and sample preparations that are disposed of to the sewer must be documented on the "Drain Disposal Log" sheets (Exhibit A).
   b) Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) may be discarded directly into the garbage. As with the water samples, all labels with the client name must be removed from the containers.

2. High Level Disposal – Samples that are hazardous waste and must be recorded on the "Waste Disposal Log" sheets (Exhibit B). An employee certified to handle the hazardous waste containers must accompany the Sample Custodian during disposal.
   a) Water Samples and Acid Digestions - the sample material is placed directly into the high-level metals waste storage container.
   b) Native Solid Samples and Dry Weights - All native solid samples (not sample preparations) may be disposed of directly into the hazardous waste container.
   c) Solvent Extracts - All solvent extracts must be treated as hazardous waste. Solvent extracts may be consolidated in clearly marked containers near the hazardous waste fume hood, and disposed of as hazardous waste.

3. Non-Routine Disposal - Samples that are designated by the client to be high level in an analyte not performed by BRL shall be considered hazardous and treated as hazardous waste upon authorization for disposal. In certain cases,

BRL may contract with a client to analyze samples that are known to be hazardous beyond the scope of our analysis (such as samples containing a high level of organic contaminants or dioxins), these samples will be flagged as requiring special disposal - by lab manager's instruction - and disposed of through a licensed hazardous waste acceptance facility. BRL may also arrange with the client to return the leftover samples to the client after analysis.

4. High Level Metals Waste Transport and Ultimate Disposal - Once a sufficient volume of waste is generated, warranting proper disposal, a waste disposal company should be contacted, and the waste scheduled for pick-up. While it is the responsibility of the waste handling company to transport and dispose of the high metal level waste in a manner consistent with local and federal environmental laws and regulations. BRL recognizes that as the generator ultimate liability can fall on BRL, and therefore every attempt is made to ensure that our contracted TSD company properly handles, transports, and disposes of all waste generated by BRL. The hazardous waste facility currently utilized by Brooks Rand is Philip Services Corp. in Renton, WA.

5. Low-level Radioactive Waste - All samples that are required to be disposed of as low-level radioactive waste need to be disposed of as detailed in SOPs BR-1600 and BR-1601 regardless of the concentrations of metals.

6. *Special Note about Biota samples and preparations:* High-level original biota samples (such as fish) must be consolidated for hazardous disposal separately from other hazardous waste. Original biota samples (such as fish, bivalves, mammal tissue, etc.) that are below disposal limits may be discarded directly into the garbage. Hazardous biota digestates (either acidic or alkaline) do not need to be composited separately, as the tissue has been degraded enough to be treated the same as other solid digestates. However, high-level biota dry weights do need to be treated as original biota samples, as the process for dry weight preparation does not sufficiently alter the tissue composition.

E. Transferring Guidelines:

1. Sample Preps – Sample preps in Teflon vials or bottles can be transferred into ultraclean 40mL glass vials or an equivalent. Transferred samples and samples in non-Teflon containers can be put in Cab #8 for long-term storage. Cab #8 should be cleared of the sample preps once a year.

2. Original Samples - Upon a client's request, solid samples may be transferred to BRL Freezers #2 for long term storage. If solid samples are in Teflon containers, and are to be transferred, the samples should first be removed from the Teflon containers and placed in acid cleaned sample jars or other appropriate non-Teflon containers. Before moving samples to Freezer #2, all of the samples for a particular project or tracking number should be placed in a bag, and the bag should be **marked clearly with the tracking number,**

'name of client and the sample receiving date. At least once a year, Freezers #2 should be cleared of any samples that are more than one year old.

7. DOCUMENTATION

Samples and preparations that are considered hazardous waste will be recorded upon disposal in the Waste Disposal Log, which is kept in a bound notebook at the sample disposal fume hood located in the shop. The "Internal Custody" sheets (SOP #BR-0301, Exhibits B and C) are used to document the disposal of samples and sample preparations as well as the transfer of samples or preparations from one location of the laboratory to another. Forms for disposed samples are filed in the back lab. Forms for transferred samples shall be kept with the samples or preparations until disposal. Special customer requirements may necessitate additional documentation, which shall be implemented as the need arises.

Drain Disposal Log for Neutralized Waters within the Acceptable Sewer Limits

Date	Initials	Volume	Notes

Brooks Rand, LLC
Waste Disposal Log

Date	Initials	Supervisor Initials	Volume Disposed	Disposal Location

COPY

SOP #BR-0304

Sample Processing

Brooks Rand, Ltd.

Written 3/16/93
Revised 6/10/02

Revision 003

Reviewed ______________________

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______________________	11/11/02
President	Date
______________________	10/22/02
QA Manager	Date
______________________	10/22/02
Senior Scientist	Date
______________________	______________________
Scientist (if applicable)	Date

Sample Processing

1. DESCRIPTION
   A. <u>Definition</u>: Documentation of the flow of sample processing from batching of samples to final review of data.
   B. <u>Scope</u>: To ensure all sample requirements are completed and documented.
   C. <u>Summary</u>: Comments for each step of sample processing will be written on a Sample Processing Form (SPF) and initialed and dated.

2. PROCEDURE

Note: Tracking of original samples and sample aliquots is achieved by SOP # BR-0306, *Sample Tracking*, # BR-0303, *Sample Storage and Disposal* and # BR-0300 *Receipt of Samples*. Together, these SOPs cover the documentation of the custody of samples from receipt to disposal including all processing for the actual analytical work. This SOP primarily covers the tracking and documentation of analytical sample processing and the stages of data review.

A. <u>Sample Batching</u> - After samples are received and logged in (see SOP BR-0300), the samples are then batched by the Laboratory Manager. Batches are sequentially numbered starting with the last two digits of the year followed by a three digit sequential number. For example the first batch in 2001 is numbered 01-001. Batch numbers are assigned to each sample on the BRL "Tracking" database. The Sample Processing Form (SPF) is then generated for each batch of samples (see Exhibit A).

B. <u>Sample Preparation</u> - The SPF is given to the scientist responsible for sample preparation. From the time the scientist removes samples from the storage area the SPF must remain with the sample batch. All sample preparation must be documented on a form or in a logbook. Copies of all preparation documentation, once complete, must accompany the SPF. The SPF must be signed and dated by the scientist performing the sample preparation, and any comments on unusual observances or deviations from the analytical SOP must be documented. The person performing the sample preparations should also update the batch status in the BRL "Tracking" database to indicate that this phase of the process is complete. After sample preparation is complete, the prepared samples, along with the SPF and preparation documentation, should be stored in the appropriate sample storage area (see SOP #BR-0303, *Sample Storage and Disposal*.

C. <u>Sample Analysis</u> - When a batch of samples is analyzed, the analyst must sign and date the SPF. Any comments on unusual observances or deviations from the analytical SOP must be documented and must be referenced on the SPF. After analysis is complete, all data including the raw instrument printouts, the analytical bench sheets, and the preparation notes must be attached to the SPF. The analyst should also update the batch status in the BRL "Tracking" database to indicate that this phase of the process is complete.

D. <u>Raw Data Review</u> - The scientist who analyzed the batch is also responsible for performing the raw data review. When the data is reviewed, the reviewer must sign and date the SPF and comment on any unusual observances or deviations from SOP # BR-1300. The data reviewer should also update the batch status in the BRL "Tracking" database to indicate that this phase of the process is complete.

E. Data Entry - Again, the scientist who analyzed the batch is also responsible for entering all raw data into the computer spreadsheets. After data entry is complete (see SOP # BR-1301), the SPF is signed and dated. Comments on any unusual observances or deviations from SOP # BR-1301 should be documented on the SPF. The computer result pages are not printed out at this time, but instead are reviewed in electronic format during the final review. The data package is then submitted to the QA Manager for final review. The person performing the data entry should also update the batch status in the BRL "Tracking" database to indicate that this phase of the process is complete.

F Final Review - The QA Manager reviews the final data (see SOP # BR-1303) and prints out the computer result pages which are then included in the data package. After final review, the QA Manager must sign and date the SPF and include comments on any unusual observations and/or deviations from SOP # BR-1303. The QA Manager should also update the batch status in the BRL "Tracking" database to indicate that this phase of the process is complete. The data packages are then stored in the "Active Client" file (located in the Project Manager's office) until all data is complete for a particular set of samples (as defined by the client) at which time the case narrative is prepared.

3. ADDITIONAL MEASURES

A. Incomplete analysis of a batch - If all samples included on one SPF are prepared together but not analyzed on the same day or under the same calibration curve, then this should clearly be noted on the SPF. In such an instance, all batch specific QA samples should be analyzed during each analysis to ensure that no degradation in the sample preparations has taken place. If the method specific preparation holding time is surpassed for the samples not analyzed, then each of the samples not analyzed is crossed out from the original SPF with a single line that is initialed and dated. The samples not analyzed are then rebatched using an identification number other than that used for the batch of which they were originally a part.

B. Deviation Traceability - The SPF and the *Analytical Non-conformance and Resolution* Form (see SOP #BR1204) are the primary documents used to record analytical deviation and/or problems. The SPF should contain any mention of unusual events or occurrences or deviations from SOPs and should list where this information can be found if relevant. Examples include, but are not limited to the following:

- Bad calibration curve-see lab data sheet.
- Samples not cold when removed from refrigerator-see instrument logbook.
- Samples over distilled-see distillation log sheet.
- Samples prepared different from SOP-see prep. notes.

In this way all necessary information concerning samples and all sample-handling steps can be traced and noted in the report to the customer.

4. QUALITY ASSURANCE

Each person is responsible for filling out the appropriate information for the task that they performed. The next responsible person will not accept the data and SPF unless the previous section is complete.

SAMPLE PROCESSING FORM

Batch # Analysis Method:

Tracking #	Lab ID	Project Ref #	Data Due Date	Matrix	Comments
QA					

Tracking # See SOW See Memo See Proj Mgr Consult MSDS See Contract Info See Lab Mgr

QA Comments

Batched By ____________ Date ____________

Prepared By ____________ Date ____________
Comments: ____________

Analyzed By ____________ Date ____________
Comments: ____________

Data Entry By ____________ Date ____________
Comments ____________

Primary Data Review B ____________ Date ____________
Comments: ____________

Final Review By ____________ Date ____________
Comments: ____________

COPY

SOP #BR-1205

Preventative Maintenance

Brooks Rand, LLC

Written 2/25/02
Revison 000

Reviewed ______________________

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______________________ President   02/28/02 Date

______________________ QA Manager   02/26/02 Date

______________________ Senior Scientist   2/26/02 Date

______________________ Lead Scientist (if applicable)   ______ Date

Preventative Maintenance

1. DESCRIPTION

A. Definition: All laboratory equipment undergoes routine maintenance that follows a clearly defined schedule. Routine maintenance is performed in a proactive manner in order to prevent problems from arising in the first place.

B. Scope: BR-1205 describes the type of maintenance to be carried out, as well as the schedule of routine maintenance for each major piece of laboratory equipment and laboratory space used at BRL.

C. Summary: Each piece of laboratory equipment undergoes routine maintenance on a daily, weekly, monthly, quarterly, semi-annual, and/or annual basis. All maintenance is documented in each equipment's instrument log and the instrument is tested prior to being used for any sample analysis.

2. EQUIPMENT

A. Equipment List:

- BRL Model III CVAFS Hg Detector
- Perkin Elmer Model 703 Flame AAS Detector
- Perkin Elmer Model 5000 GF and Flame AAS Detector
- Perkin Elmer Model 4100ZL GF ZAAS Detector
- Sequoia-Turner Model 690 Colorimetric Spectrophotometer
- Perkin Elmer Model 1420 IR Spectrophotometer
- Orion Research Model 301 pH Meter
- VWR Pure H_2O Tester Conductivity Meter
- Mettler Model H10 Analytical Balance (to 0.1 mg)
- Sartorius Model E1200 Top Loading Balance (to 1 mg)
- ACCULAB Model VI-350 Top Loading Balance (to 10 mg)
- Mettler Model BB2400 Top Loading Balance (to 10 mg)
- Various pipettes
- Oven and Freezer Thermometers
- Omega Model CN76000 Thermocoupler
- Fisher Scientific Model 630F Scientific Oven
- Labline Instruments Isotemp Scientific Oven
- Forma Scientific Model 3927 Incubator/Refrigerator
- Sears Coldspot Refrigerator Freezer
- Kenmore Model 20 Refrigerator/Freezer
- General Electric Model CB22D Chest Freezer
- Sears Coldspot Chest Feezer
- Montgomery Ward Model FFT-4945 00L Upright Freezer

B. Various instrument logbooks

3. PROCEDURE

All laboratory equipment undergoes scheduled routine maintenance to ensure that it is functioning properly prior to being used for sample preparation or analysis. Each instrument has its own "instrument log" where all routine checks and maintenance are documented (refer to SOP #BR-1200). A copy of the overall lab maintenance schedule is presented as Exhibit A. The following sections break the schedule down into the various instrument groups utilized at Brook Rand LLC.

All listed maintenance is the minimum required. Other non-routine maintenance may be required as dictated by system performance.

A. Trace Level Mercury System (CVAFS): The trace level mercury system is located in the Clean Room Lab and it includes both instruments setup to analyze for total mercury (BRL Model III Hg Detectors #s BR-06 and BR-08) and the instrument setup to analyze for methyl mercury (BRL Model III Hg Detector #BR-05. The frequency of maintenance can be broken down into daily, weekly, monthly, quarterly, semi-annual, and annual schedules.

1. Daily: Prior to each analytical run.
   a. Check all fittings to ensure that they are secure and not leaking.
   b. Rinse and refill the DDIW bottle.
   c. Prepare new pretraps for total mercury analyses.
   d. Blank each analytical trap prior to analysis.
   e. Blank each analytical trap after each non-zero run for the methyl mercury system.

2. Weekly: Friday of each week.
   Soak bubblers over the weekend in 1% KOH solution.

3. Monthly: The last Friday of each month.
   a. Inspect all tubing for signs of wear.
   b. Replace any loose fittings.
   c. Prepare new stock standards.

4. Quarterly: The last Thursday/Friday of January, April, July, and October.
   a. Make, test, and change out traps on the total mercury system.
   b. Condition the GC column on the methyl mercury system. The column should be heated at 180° C overnight (Thursday afternoon to Friday morning). Heating the column over the weekend could significantly reduce the column's efficiency.

5. Semi-Annual: The last Friday of January and July.
   a. Replace lamps.
   b. Make, test, and change out traps on the methyl mercury system.
   c. Blank traps on the incoming gas lines (replace as needed).

6. Annual: The last Friday of January
    a. Clean/change the quartz cell.
    b. Replace GC column on the methyl mercury system.
    c. Ensure that there are backup analytical traps, mercury lamps, and Teflon tubing.

B. <u>Hydride System (HGAAS) – Perkin Elmer Model 703</u>: The hydride system is located in the AA Lab. The maintenance schedule is as follows.

1. Daily: Prior to each analytical run.
    a. Rinse and refill the DIW bottle.
    b. Check all fittings to ensure that they are secure and not leaking.
    c. Rinse water removal trap with DIW at the end of the analytical day.

2. Weekly: Friday of each week.
    a. Soak bubblers over the weekend in 1% KOH solution.
    b. Clean the spectrophotometer windows.

3. Monthly: The last Friday of each month.
    a. Inspect all tubing for signs of wear.
    b. Replace any loose fittings.
    c. Prepare new stock standards.

4. Quarterly: The last Friday of January, April, July, and October.
    a. Ensure that there are working traps for both arsenic and selenium.
    b. Ensure that there are backup traps for both arsenic and selenium.

5. Semi-Annual: The last Friday of January and July.
    a. Verify that there are working backup lamps for both arsenic and selenium.
    b. Clean nebulizer.

6. Annual: The last Friday of January
    a. Fine tune the instrument wavelength.
    b. Check instrument optics.
    c. Test backup lamps.

C. <u>Graphite Furnace System (GFAAS) – Perkin Elmer Model 4100ZL</u>: The graphite furnace system is located in the AA Lab. The maintenance schedule is as follows.

1. Daily: Prior to each analytical run.
    a. Check graphite tube.
    b. Clean furnace housing.

2. Semi-Annual: The last Friday of January and July.
    a. Change graphite contact cylinders.
    b. Document availability of lamps for each analyte.

3. Annual: The last Friday of January
   a. Test lamps (over several days) for all analytes.
   b. Ensure that all lamps are identifiable by a unique serial number so that test results can be traced back to each lamp.

D. Mercury System (CVAAS) – Perkin Elmer Model 5000: The mercury system for the analysis of samples by EPA Method 245.1 is located in the AA Lab. The maintenance schedule is as follows.

Daily: Prior to each analytical run.
1. Clean instrument.
2. Verify that instrument in working order.
3. Check supply of chart recorder paper.

E. Deionized Water System:

1. Daily: Prior to use.
   a. Test conductivity of DIW/DDIW at source in each lab.
   b. Check system pressure differential and change filters as needed. **Always** change filters on a Friday to allow the system to stabilize over the weekend.

2. Weekly: Friday of each week.
   Check probe cables on conductivity meter.

3. Monthly: The last Friday of each month.
   Check calibration of conductivity meter.

4. Semi-Annual: The last Friday of January and July.
   Change 0.2 µm DDIW final filter.

5. Annual: January
   a. Schedule US Filter to change out the carbon filter.
   b. Schedule US Filter to recharge the mix bed tanks.

F. Balances:

1. Daily: Prior to use.
   Check calibration over range of weights being used.

2. Monthly: The last Friday of each month.
   Conduct a four-point calibration check for each balance.

3. Annual: February
   Schedule certified calibrations for each scale.

G. Thermometers:

1. Monthly: The last Friday of each month.
   a. Check thermometers for wear.
   b. Check thermal couplers on sand baths for wear.
2. Annual: October
   a. Schedule certified calibrations for NIST certified thermometers:
      i. VWR 61054-546 refrigerator/freezer thermometer
      ii. VWR 61222-548 oven thermometer
   b. Check calibration of all other thermometers and temperature controlling devices against the NIST certified thermometers.

H. Ovens/Refrigerator/Freezers:

1. Daily: When in use.
   Check and record the temperature in all ovens, refrigerators, and freezers.

2. Weekly: Tuesday of each week.
   a. Change temperature recorder paper in Refrigerator #2 and Freezer #4.
   b. Check recorded temperatures for anomalies and report accordingly.

3. Monthly: The last Friday of each month.
   Clean the interior of each oven, refrigerator, and freezer.

4. Semi-Annual: Last Thursday-Friday of January and July
   Defrost BRL Refrigerator #3.

5. Annual: Last Thursday-Friday of January
   Defrost all other refrigerators and freezers as needed.

I. General Lab Cleanliness:

1. Daily:
   Restock shoe cover bins in Hg (Clean Room) and AA Labs.

2. Weekly: Friday of each week.
   a. Wipe all surfaces with a clean, damp rag to remove dust and chemical residues from all lab spaces.
   b. Empty all recycling and garbage bins.

3. Monthly: The last Friday of each month.
   a. Clear, dispose, and store all clutter.
   b. Monitor air for Hg concentration in clean hood of the Hg Lab, by the door of the Hg Lab, and in Receiving and the Prep (Down) Lab.
   c. Wipe inside fume hoods (all walls and benches) located in all labs.

J. Miscellaneous Equipment/Supplies:

1. Pipettes: The calibration must be checked at the user volumes for each pipette prior to use. Monthly, all pipettes must be calibrated over the range of volumes for which they are used.

2. pH Meter: A two-point calibration should be performed prior to use and the pH meter must be cleaned after each use.

3. Spectrophotometer: The sample compartment and windows must be cleaned prior to and after each use. Lamp alignment and instrument electronics should be checked annually.

4. Laminar Flow Hoods: The prefilters of the flow hoods should be changed quarterly. The Hg removal filters (iodated carbon) should be changed annually, or as needed.

5. Acid Vats: The acid in the vats used to clean bottles and lab ware must be analyzed for mercury concentration at least monthly. Additionally, the temperatures of the vats must be verified monthly.

6. Acids/Reagents: All supplies should be checked weekly and ordered as needed. New acids must be analyzed for Hg concentration prior to use. Reagents used for the analysis of total mercury by EPA Method 1631 must be analyzed for Hg concentration monthly, and when prepared prior to use.

7. Gases: All supplies should be checked weekly and ordered as needed. The air compressor valve must be opened and excess water allowed to drain every other month.

8. Computer Files: All Access and PO databases, QA files, and lab files must be backed-up to CD-ROM on a weekly basis.

4. QUALITY ASSURANCE

A. Monthly Checklist: The QA Manager is responsible for electronically producing a monthly schedule that outlines all of the preventative maintenance required for the upcoming month and specifies who is to complete each task. The schedule contains check-off spaces for each task and room for any necessary comments. Each responsible party is required to enter when they completed each task. At the end of the month, the QA Manager reviews the past month's schedule. Any uncompleted tasks are noted in the QA Managers monthly report and are forwarded to the next month's schedule as necessary.

B. Internal Audits: the QA Manager performs monthly audits of the laboratory spaces and equipment. All equipment logbooks are checked during these audits to ensure that all equipment performance and maintenance is being properly documented for each instrument.

Exhibit A

Analytical Lab Maintenance Schedule

ITEM	Daily[1]	Weekly	Monthly	Quarterly	Semi-Annual	Annual
Acid Vats			collect & analyze acid for Hg concentration, check temperature			
Balances	*calibration check*[2]		*four-point cal. check*[2]			*certified calibration*[2]
Pipettes	*calibration check at set volume(s)*		calibrate over range of settings			
Safety Check		flush eyewashes (5) & showers (2)	*inspection (CHP app. D)*			certify fire extinguishers
Thermometer Calibration			check thermocouplers for wear			check using NIST-*certified therm.*[2,3]
Temperature Checks	ovens as used, freezer, refrig.[2]	change temp. recorder paper in Ref.#2 & Frz.#4	clean interior of ovens, freezer, refrig.[2]			defrost freezers as needed
Acids/Reagents		check supply, order & test if necessary				
BR-05 CVAFS & MMHg system	rinse/refill DDIW bottle; check fittings; blank traps prior to analysis and after each nonzero	soak bubblers in 1% KOH; blank traps	change loose fittings; prepare new stock standards	*condition GC column*	replace traps; replace *lamp; blank traps on* gas lines	clean/change quartz cell; replace GC column
BR-06 CVAFS & T-Hg system	rinse/refill DDIW bottle; check fittings; prepare new pretraps; blank traps prior to analysis	soak bubblers in 1% KOH; blank traps	change loose fittings; prepare new stock standards	replace traps	replace lamp; blank traps on gas lines (replace as needed)	clean/change quartz cell
BR-08 CVAFS & T-Hg system	rinse/refill DDIW bottle; check fittings; prepare new pretraps; blank traps prior to analysis	soak bubblers in 1% KOH; blank traps	change loose fittings; prepare new stock standards	replace traps	replace lamp; blank traps on gas lines (replace as needed)	clean/change quartz cell
Hydride AA (PE 703)	rinse/refill DIW bottle; check fittings; rinse water removal trap	clean spectroph. windows; soak bubblers in 1% KOH	change loose fittings; prepare new stock standards	ensure working traps for As, Se & one backup set	verify backup lamp available; clean nebulizer	fine tune wavelength; check optics; test backup lamps
Hg 245.1 AA (PE 5000)	clean, verify all equip. in working order, check *supply of chart paper*					

tracking number 02BR001). The samples within a shipment are then each identified by a two digit sequential number. For example, if three samples were received in the 02BR001 shipment they would be given the sample numbers 02BR001-01, 02BR001-02 and 02BR001-03. On the sample receiving log the client's sample ID and the BRL ID numbers are listed for each sample. The BRL tracking numbers (not the client's ID numbers) are referenced during all laboratory preparations and analyses.

Fluoropolymer sample bottles are all engraved with a bottle ID number that is used for sample identification purposes. Clients may wish to use additional sample identification numbers but the unique sample container numbers must be documented on the Sample Receiving Log. When a bottle is removed, the number engraved on the bottle should be matched with the number written on the bag. Any discrepancies should be noted in the Sample Receiving Log. Each bottle should be rinsed with clean DI water (for low-level samples) and/or wiped with a clean cloth. Bottles are then placed in the clean hood, and labeled with the BRL sample number, customer project number, date of sample receipt, client name, and sample preservation information. An example of a BRL sample label is as follows:

02BR450-01 CWP001 4/11/02
City Wastewater Treatment Plant
0.5% BrCl

The original BRL Sample Receiving Log sheets must be signed by the Sample Custodian (or designated alternate) and are kept in a three ring binder at the sample receiving desk. After Sample Receiving Log sheets accumulate up to 100 tracking numbers, the Sample Custodian should bind the originals in a velo-binder and give the bound receiving sheets to the QA Manager for archival.

In addition, an "Internal Custody for Original Samples" form is generated at the time of sample log-in. This form documents the life cycle of each sample shipment from receipt to disposal, and is kept with the samples at all times. After samples have been disposed, the custody forms are stored in the Sample Custodian's files.

8.2.4 Sample Storage

All samples are stored in a secure area. A secure area is defined as a locked area within the premises of BRL with restricted access. To satisfy these custody provisions, the laboratory implements the following procedures:

- Access doors to the laboratory are kept locked, except during normal working hours
- Visitors must sign in and are escorted while in the laboratory
- Samples remain in the secure area until they are removed for sample preparation or analysis

Brooks Rand Ltd.   Chain of Custody Record

Client:	e-mail address:	Ship to: **Brooks Rand Ltd.**
Contact:	PO #:	3950 6th Avenue NW
Address:	Sampler's signatures:	Seattle, WA 98107
		206.632.6206
		206.632.6017 fax
Phone #:	Client project ID:	e-mail: brl@brooksrand.com
Fax #:	BRL project ID:	www.brooksrand.com

For BRL use only	**Cooler temp (°C):**	**Custody seals present? (Y/N)**	**Custody seals intact? (Y/N)**	**Date:**	**Initials:**

	Sample ID	**Collection**		**Miscellaneous**				**Field Preservation**					**Analyses required**							**Comments**
		Date	Time	Sampler (initials)	Matrix type	# of containers	Sample field filtered , Y/N	Unpreserved (ice only)	HNO_3	HCl	BrCl	Other (specify)								
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				

Shipping carrier:	# of coolers:

Relinquished by:	Date:	Time:	Received by:	Date:	Time:
Relinquished by:	Date:	Time:	Received at BRL:	Date:	Time:

White: LAB COPY   Yellow: CUSTOMER COPY

CUSTODY SEAL

Date ______________________________

Signature ____________________________

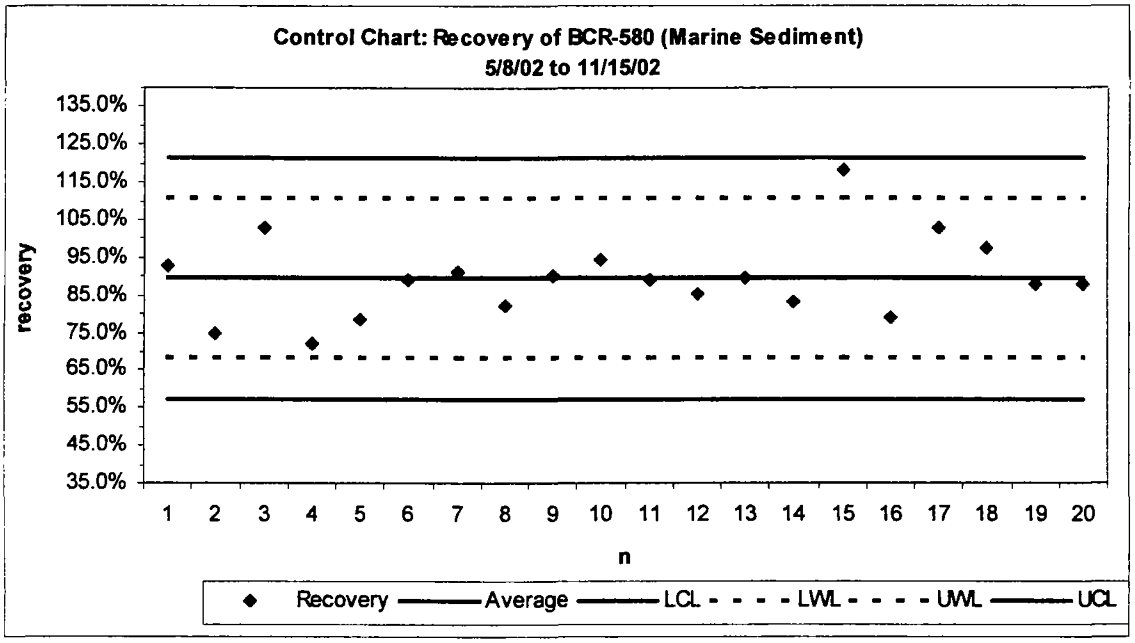
I-CHEM®
Brand Products

Nalge Nunc International

90009

Brooks Rand – Methyl Mercury Control Charts (BR-0011), Sediment/Soil

Accuracy



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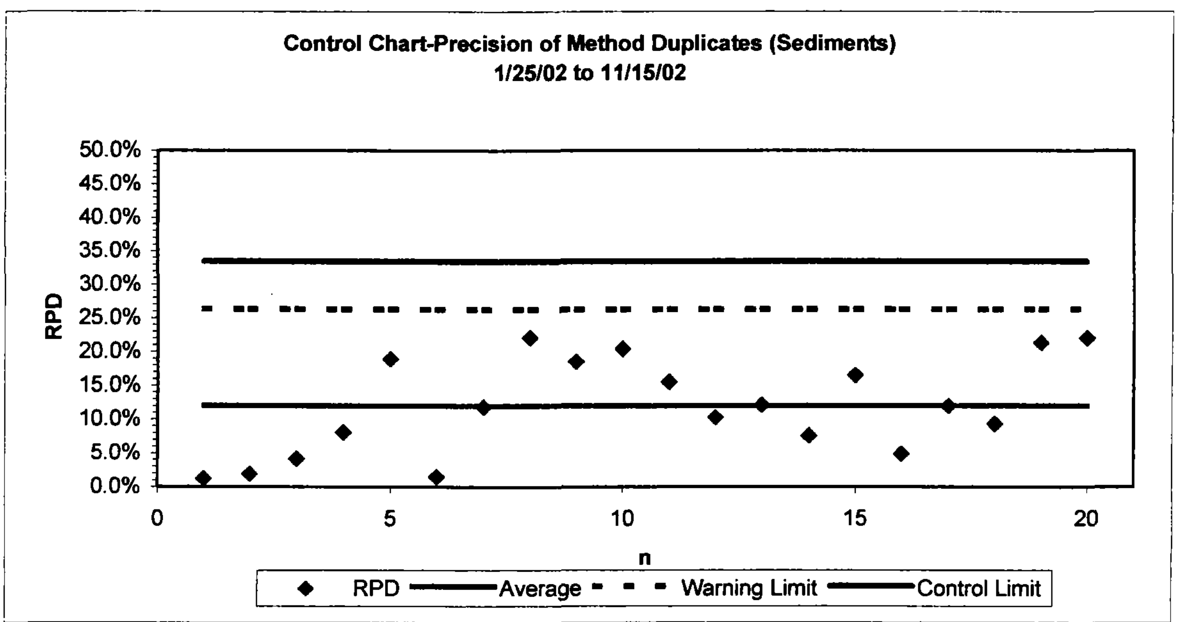
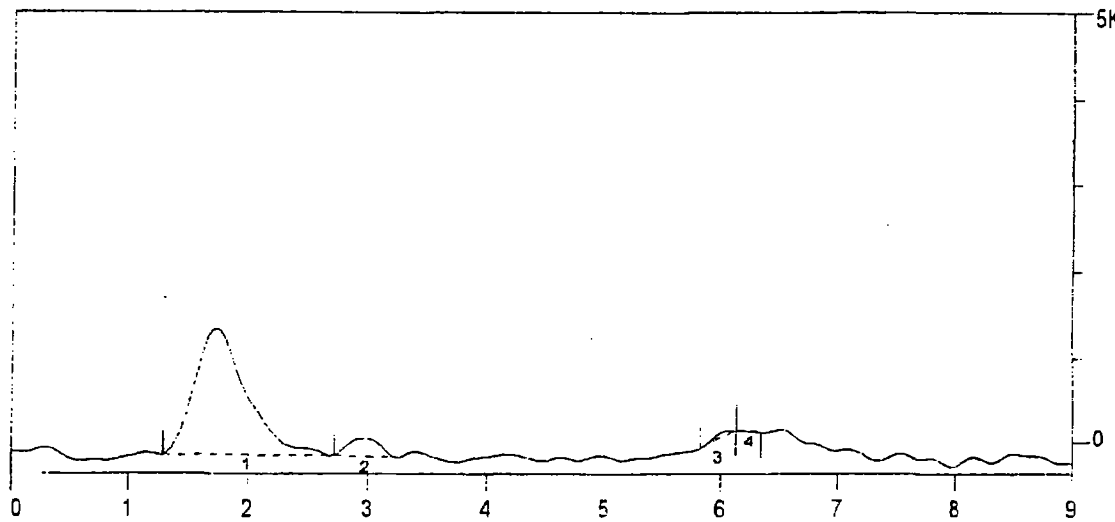


Brooks Rand LLC

Monomethyl Mercury Chromatograms



Feb 11 2003
10:37 AM

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2	2.97	39 593
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4	6.16	645

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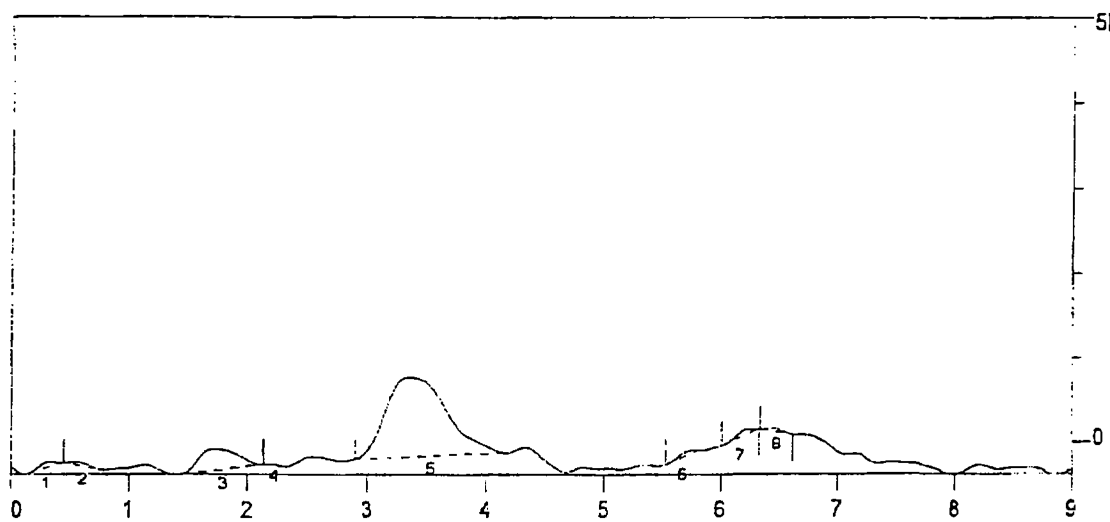
Analyst	t johnson
Tracking	cal blank
Notes: 03-012-1	



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10:48 AM

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6	5.76	4 786
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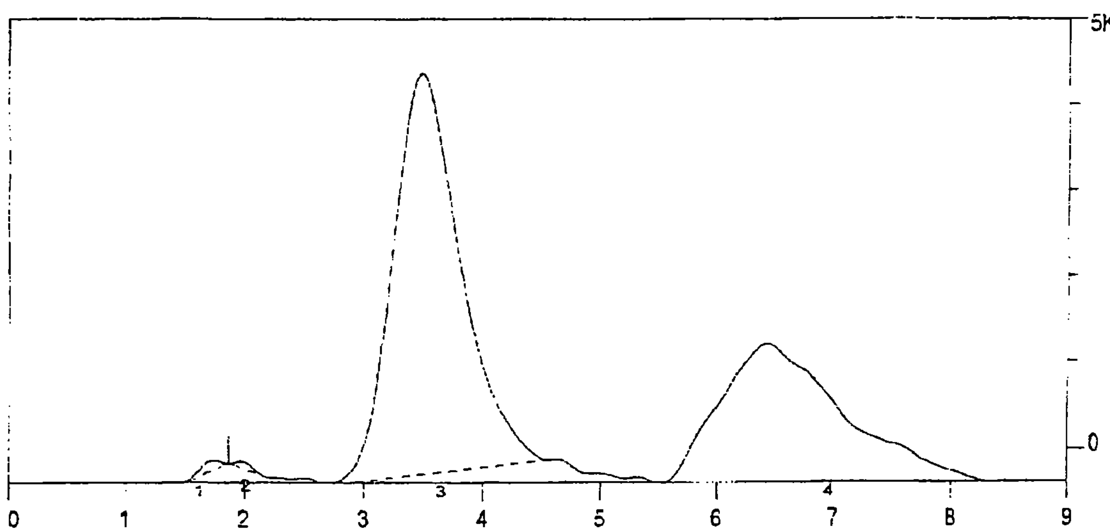
Tracking	10pg cal std
Notes: 03-012-1	



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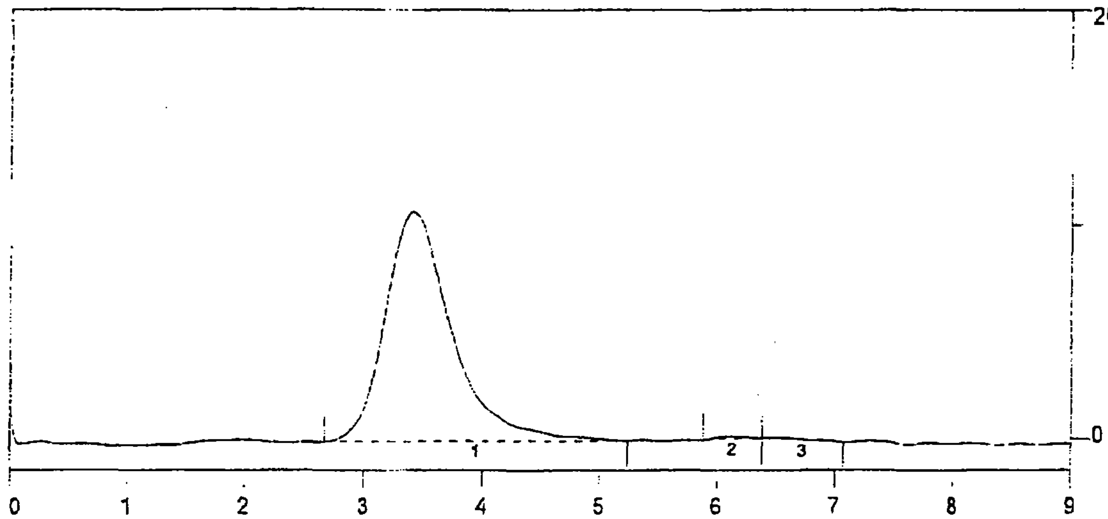

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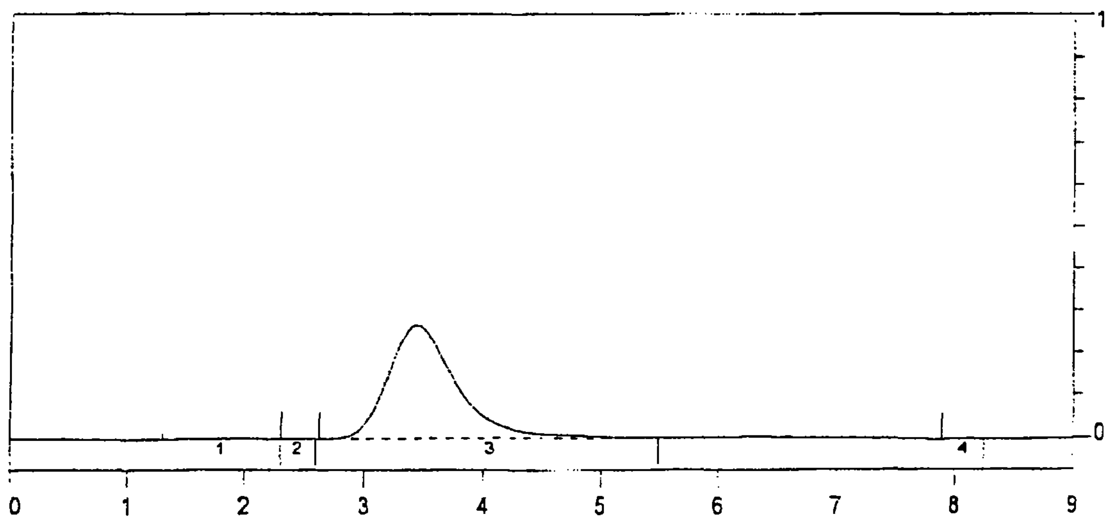

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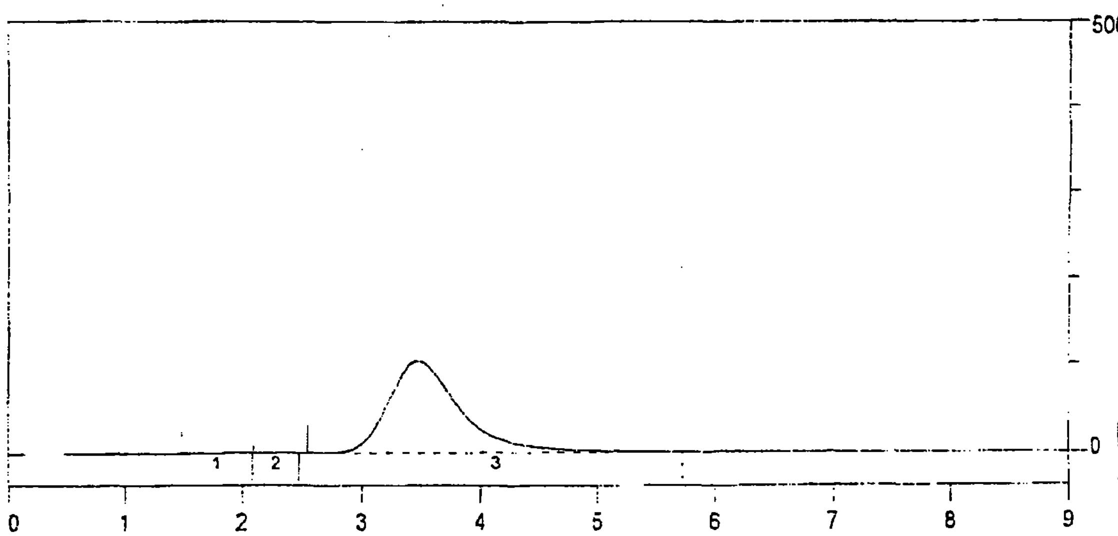

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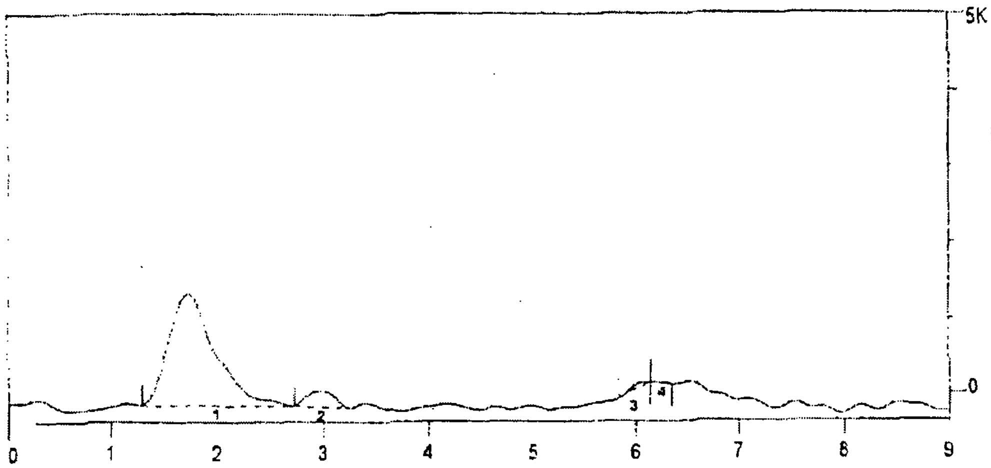
Analyst	t johnson
Tracking	1000pg cal std
Notes: 03-012-1	

Brooks Rand LLC

Monomethyl Mercury Chromatograms



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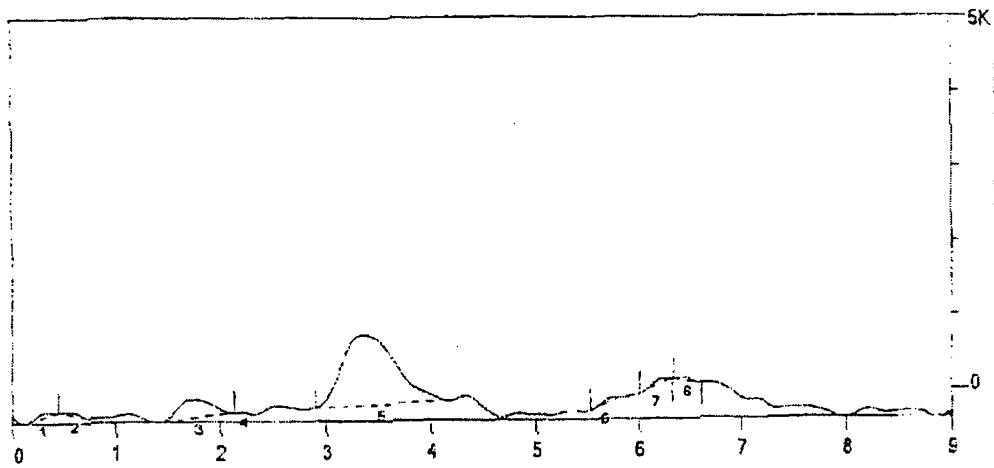

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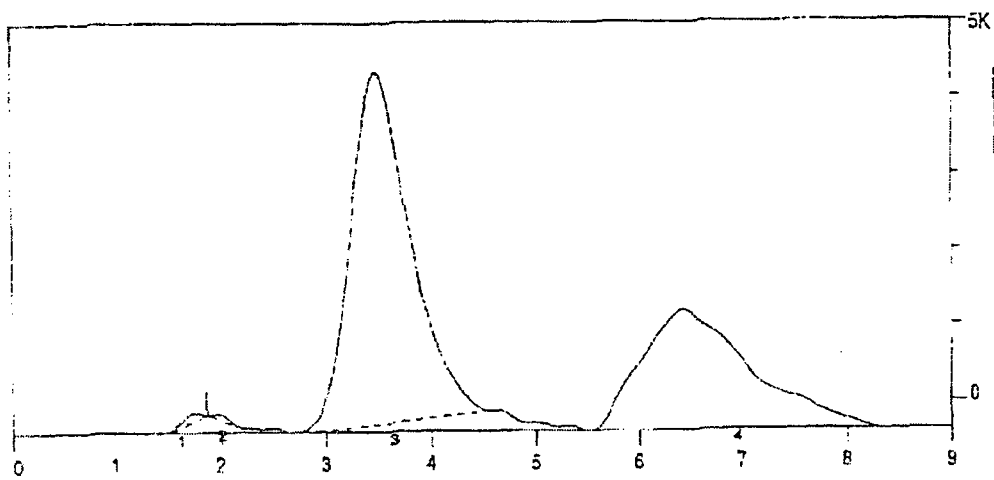

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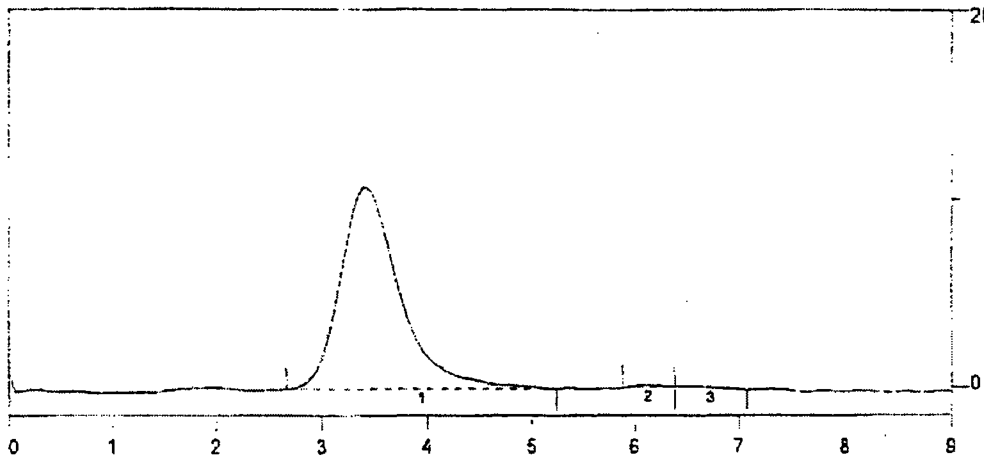

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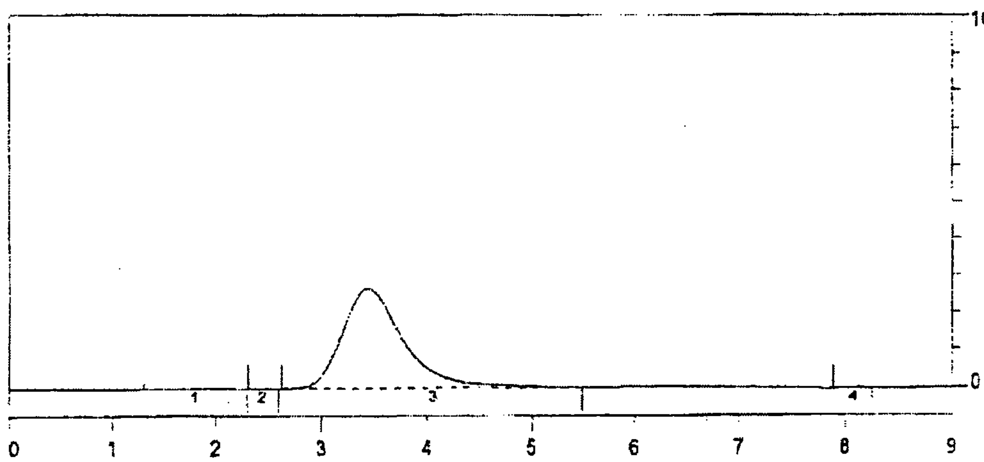

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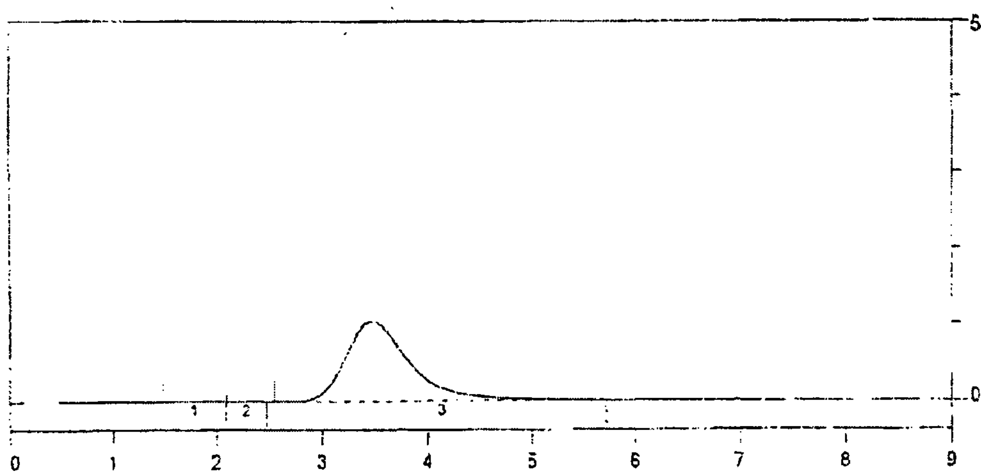

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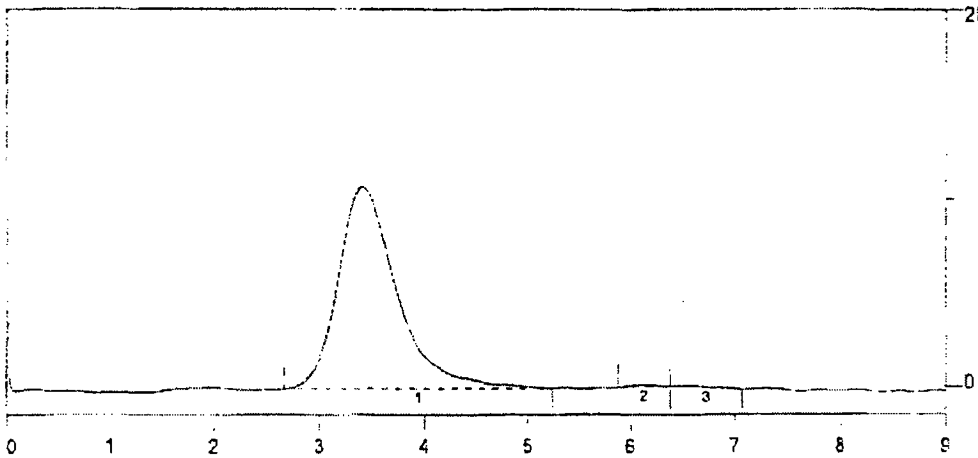

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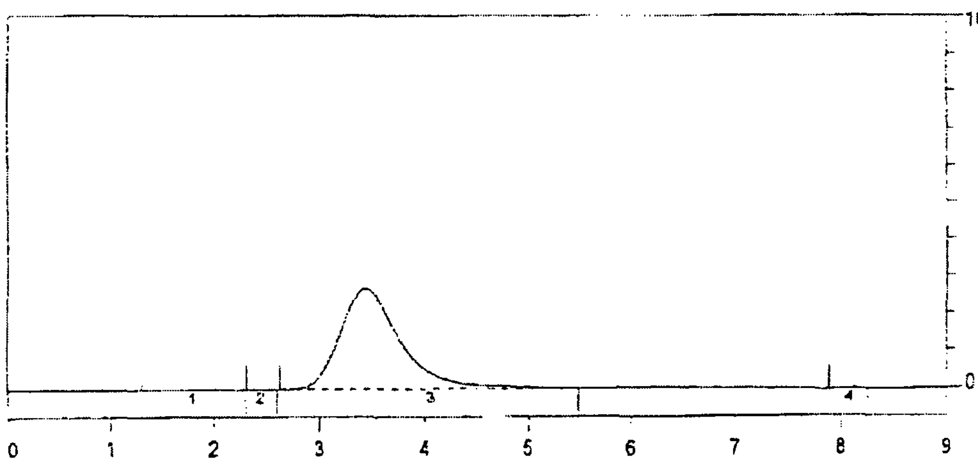

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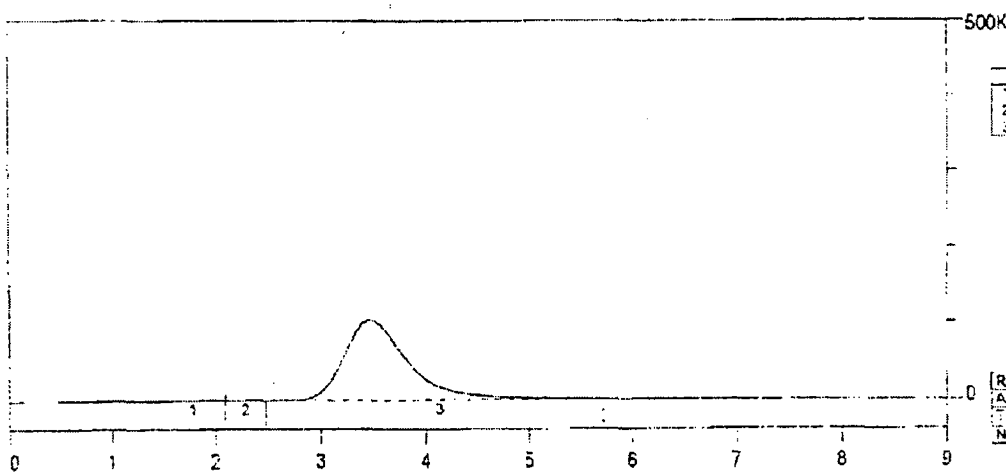

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Tracking	1000pg cal std
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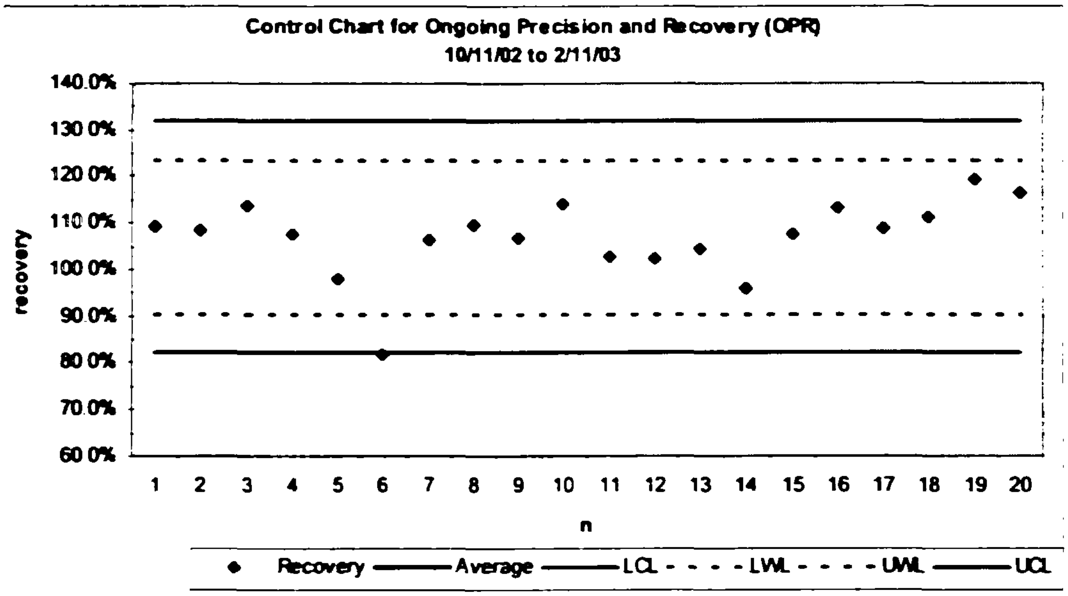
Mercury Guru ver. 2.1 © 2001 Brooks Rand Ltd.

Brooks Rand LLC
Control Chart Data
Methyl Mercury In Sediments

Ongoing Precision and Recovery



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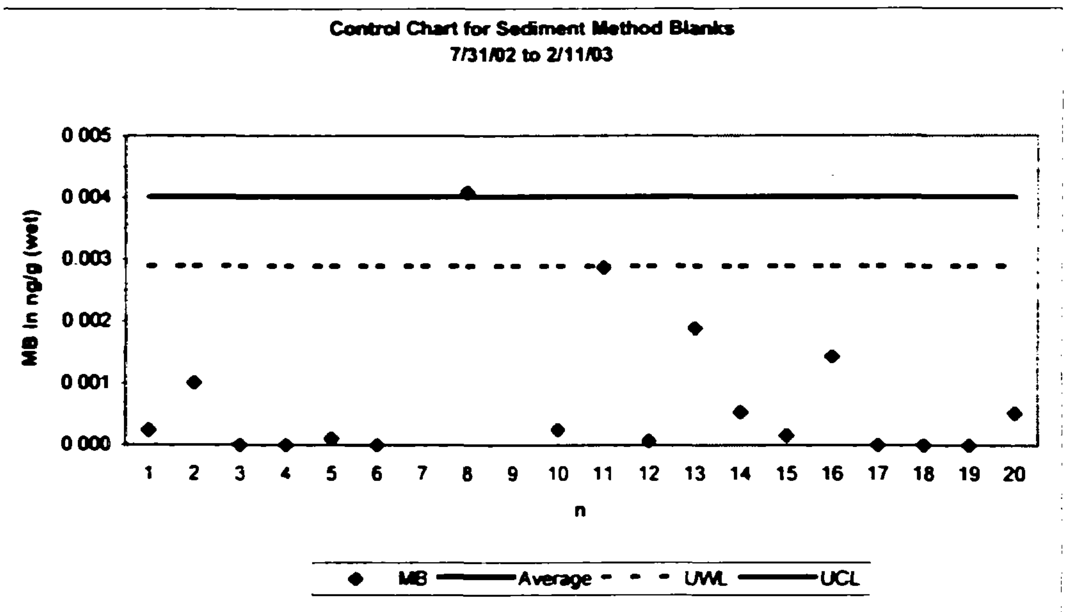

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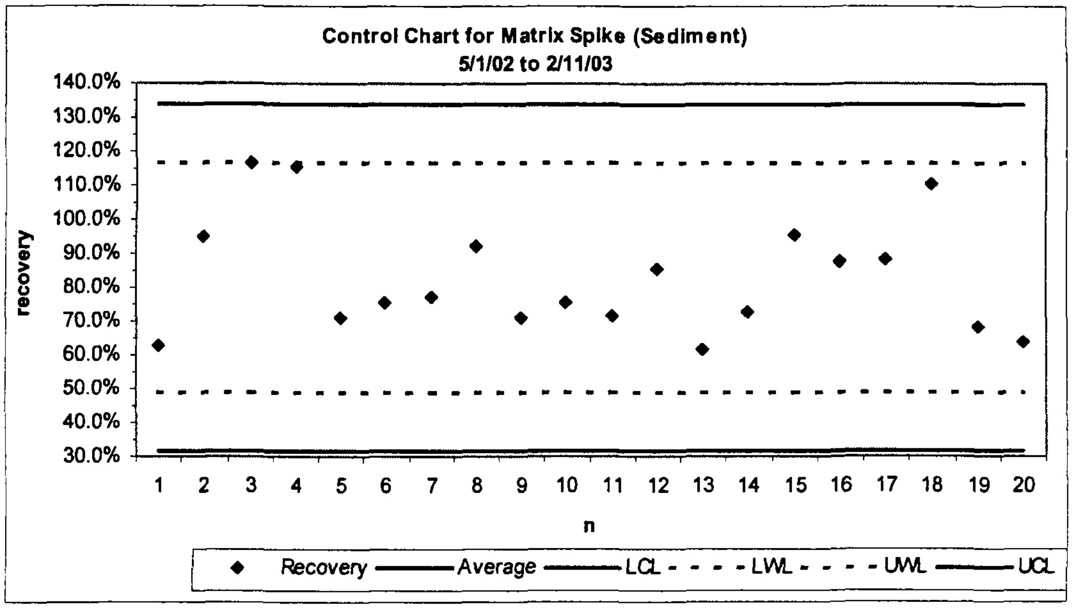

SOLID: **(BR-0011)** QUALITY CONTROL CRITERIA FOR THE ANALYSIS OF **METHYL MERCURY** BY CVAFS (ANALYSIS OF PREPS EQUIVALENT TO EPA 1630)

QC Sample	Measure	Minimum Frequency	Criteria	Corrective Action
Ethylation Blank	Contamination from bubblers	4 per batch; following each OPR	≤ 2 pg	Clean and test bubblers until criteria met prior to any analysis
Ongoing precision and recovery (OPR)	Accuracy	At beginning and end and 1 per 10 sample preparations	Recovery = 67 – 133%	Correct problem and reanalyze OPR. If criteria met, reanalyze samples backwards until 2 consecutive results w/RPD ≤ 20%
Carryover Check Ethylation Blank	Contamination due to carryover in the bubbler/trap	Following any unusually high result. Currently ≥ 2x the high standard	≤ 2 pg	Clean and continue to test bubbler/trap combo until criteria met prior to further use. Reanalyze samples that were analyzed in same bubbler/trap following high result
Method Blank	Contamination from reagents, lab ware, etc.	3 per batch	avg < MDL or < 1/10th associated samples	Correct problem. All samples associated with a contaminated method blank must be reanalyzed.
Certified Reference Material (CRM)	Accuracy	1 per batch	Recovery = 65 – 135%	Correct problem prior to continuing analysis
Matrix Spike/Spike Duplicate	Accuracy and Precision within a given matrix	1 per 10 client samples	Soil Rec=60-120%; RPD ≤ 35% Biota Rec=75-135%; RPD ≤ 35%	If recoveries similar but fail recovery criteria, an interference is present in the sample and the result must be qualified. If RPD criteria not met, then the system is not in control. Correct problem and reanalyze all associated samples.